BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JANUARY 7, 1927.

I.—GENERAL; PLANT; MACHINERY.

Theory of the absorption tower. R. C. Cantelo (Chem. Met. Eng., 1926, **33**, 680—681).—In a counter-current gas scrubbing tower where the velocities of the gas and liquid are uniform it is shown that the partial pressure of the solute in the liquid at a point x is given by $p = 1 \cdot 314T\,m/M$, where T is the average temperature in the tower, m the concentration of solute in the gas in lb./cub. ft., and M the mol. wt. of the solute. Further, $dx/dt = M.U/1 \cdot 314\,rAT' \times (1 - p'w - p')/[M/1 \cdot 314T\,(1 - pw) - m]$ where U is the volume of gas in cub. ft./hr., r is the fraction of free space per unit vol., A the cross-section of the tower, T' the absolute temperature at the top, p' the partial pressure of the solute at the top, $p'w$ the vapour pressure of the liquid at T'. These equations are further developed in order to determine the rate of dissolution dm/dt or the dissolution coefficient for the following special cases (1) in which the solute obeys Henry's law, (2) in which the solute obeys Raoult's law, as in the scrubbing of coal gas for benzol, (3) in which a chemical reaction takes place yielding a compound of negligible vapour pressure, or the solute is very soluble in the liquid. C. Irwin.

Patents.

Sectional retort for retort kilns. R. S. Kent (U.S.P. 1,602,678, 12.10.26. Appl., 6.1.21).—The kiln housing includes a combustion chamber and a heating chamber, separated by a side wall provided with a port for the passage of the combustion gases into an upper zone of the heating chamber. A vertical retort formed in separable upper and lower sections is mounted in the heating chamber. A transverse wall near the upper end of the upper retort section closes the upper end of the heating chamber, and a similar wall near the upper end of the lower retort section is provided with an opening affording communication between the upper and lower zones of the chamber. H. Holmes.

Settling tanks. S. Hunter (E.P. 260,081, 14.9.25).—The sediment is discharged from the bottom of a conical tank while preventing escape of the supernatant liquid by the use of a central rotary displacer arranged to descend by gravity. Such displacer may be a radial-bladed stirrer with the outer edges of the blades parallel to the tank walls. Its weight is so adjusted in relation to the density of the sediment that it rests on the top of the sediment as the latter descends. The sediment is cut into progressively outwards parallel with the tank walls, so that the loosened material passes inwards and downwards to the point of discharge. H. Holmes.

Separating immiscible liquids. Sharples Specialty Co., Assees. of L. D. Jones (E.P. 233,333, 21.4.25. Conv., 1.5.24).—Oil containing water (or a similar mixture of liquids) is taken from a storage tank (*e.g.*, double bottom of a ship) to a settling tank, and the watery fraction, or, if this has first been separated by gravity, the oily layer from the watery fraction, is centrifuged. The oil from the centrifuge is returned to the settling tank, but not to the storage tank. If water is in excess in the original mixture, the process is varied somewhat. B. M. Venables.

Separation of liquids. H. S. Hele-Shaw and A. Beale (E.P. 260,045, 22.7.25 and 27.7.25).—The separation of two liquids (*e.g.*, oil and water) which differ in sp. gr. and viscosity is effected in a number of substantially horizontal channels, usually quite large in number, so that the cross-section of each individual channel may be small enough to ensure stream-line flow. The upper surface of each channel is provided with perforations through which the lighter liquid rises to a collecting pipe or reservoir, which is subdivided by vertical partitions to keep the lighter liquid quiescent. The perforations may be covered with wire gauze, and interceptors of wire mesh or perforated metal may be provided in the main channels. B. M. Venables.

Liquid purification systems. The Sharples Specialty Co., Assees. of P. T. Sharples and L. D. Jones (E.P. 252,173, 2.4.25. Conv., 2.4.24).—A liquid is freed from immiscible impurities in a centrifugal separator, and, while in a highly dispersed condition in the separator, is subjected to a stream of gas to remove miscible or dissolved impurities. The apparatus may be provided with self-contained vanes to impel the gas. (Cf. E.P. 204,047; B., 1924, 9.) B. M. Venables.

Recovery of solids from their solutions. W. H. Dickerson (U.S.P. 1,600,503, 21.9.26. Appl., 12.11.21).—Liquids which at some stage of concentration become sticky, viscous, or syrupy, are converted into dry glazed particles by spraying them into the hottest part of a current of heated drying gas and permitting the liquid-laden gas to pass through a drying chamber. The intermixture is maintained for a time sufficient to permit formation of the glazed particles, which are then separated from the spent gas. H. Holmes.

Separation [of suspended matter from fluids] and apparatus therefor. C. G. Hawley (U.S.P. 1,600,762, 21.9.26. Appl., 28.6.26).—A vortex flow of the fluid, maintained by a tangential delivery, is established about an axis transverse to the main flow. The purified fluid and the suspended matter are separately discharged outwards from the vortex, in the transverse plane of the tangential delivery. H. Holmes.

Filtering apparatus. I. G. Perrett and A. J. Simpson (E.P. 259,751, 2.10.25).—A number of easily

and separately detachable filter units are attached to a common draw-off pipe and the whole is immersed in a tank containing the liquid to be filtered. The filter units are made up in cartridge form of pervious materials, and it is possible to change them one at a time without stopping the filtration through the other units, a valve for each unit being provided in the draw-off pipe, which must be closed before the unit can be removed.

B. M. Venables.

Apparatus for cooling or otherwise treating liquids with gases or gases with liquids. F. C. Smith (E.P. 259,882, 10.7.26).—The liquid passes down a cell comprising two perforated sheets, with the space between filled with metallic or other pervious material. The gas passes several times transversely to the cell, and is prevented from entraining drops of liquid by means of upwardly inclined plates at the places where the gas leaves the cell. B. M. Venables.

Treatment of hot substances which must remain excluded from the action of the air until they reach a low temperature. J. Sommermeier (E.P. 244,109, 3.12.25. Conv., 3.12.24).—The material from the heating furnace passes straight into a stream of inert gas by means of which it is conveyed to a sorting device, which deflects the coarse material through a valve to one closed bin, and allows the gas and fine material to pass to a cyclone or other separator, where the fine material is collected and drops to another closed bin. The fine material packs close, and will not usually need further cooling, but air tubes may be inserted through the bin. The gas is withdrawn from the cyclone by a fan, then passes through a cooler, and is blown through the mass of the coarse material to effect further cooling, returning to the point where the material is picked up. A relief valve is provided in the circuit, and reacting substances may be added to the gas stream. B. M. Venables.

Evaporator. C. W. Aiken (U.S.A. 1,600,784, 21.9.26. Appl., 17.5.23).—Two evaporators and two separators are provided with fixed connexions and valves arranged for the passage of steam through either the first evaporator and its associated separator in series, and then similarly through the second evaporator and separator, or through the second evaporator and separator in series and then through the first evaporator and separator. H. Holmes.

Multiple-effect distilling apparatus. G. & J. Weir, Ltd., and J. G. Weir (E.P. 259,747, 1.10.25).—The several chambers of a multiple-effect evaporator are so constructed that the area of exposed liquid and the vapour space increase in proportion to the volume of vapour generated in each effect, whilst the temperature range allowed in each effect decreases as the pressure drops, but the heating surface is increased in inverse proportion to the temperature range. B. M. Venables.

Separating mixtures of gases and vapours into fractions of different molecular weight. S. P. Szczepanowski (E.P. 237,617, 22.7.25).—The gases are subjected to gentle centrifugal action (or to gravity), and the pressure potential (*i.e.*, increase of partial pressure of the larger molecules) thus produced is utilised to separate the mixture into various fractions.

B. M. Venables.

Apparatus for drying and cooling gases, preferably for liquefying gas. A. Seligmann (U.S.P. 1,599,681, 14.9.26. Appl., 21.2.24. Conv., 26.5.22).—The high-pressure gas is passed through a valve and upwards through a conduit within a counter-current heat exchanger, which is provided with an inlet at its upper end and with valved outlets, one at its upper and the other at its lower end, for the low-pressure gas, these outlets being in communication through the heat exchanger, so that a thawing medium introduced at the lower outlet passes through the heat exchanger in the same direction as the high-pressure gas. H. Holmes.

Heating or cooling apparatus for air or other gases. J. Forgan-Potts (E.P. 260,357, 30.7.25).—The gas is blown over a nest of tubes bent into circles and provided with gills or other means of increasing the surface. B. M. Venables.

Composition for hydrometric units etc. C. E. Linebarger (E.P. 259,409, 16.11.25).—Acid-proof vessels, floats, etc. are constructed by moulding a mixture of chlorinated naphthalene (or other hydrocarbons) and stearic acid in about equal parts. A wax such as carnauba may be added to increase the hardness and smoothness, and varnish gum to increase the toughness and plasticity. Wax does not much affect the sp. gr. of the composition, but inert materials such as asbestos, talc, mica, may be added if a higher sp. gr. is desired, also colouring matter if desired. A composition of d 1·2 and m.p. 77° is particularly claimed for battery acid hydrometers. B. M. Venables.

Analysing gases [especially flue gases for carbon dioxide]. M. J. E. Chopin (E.P. 250,248, 31.3.26. Conv., 3.4.25).—A current of the gas is caused to flow at a relatively high speed in a serpentine course above the surface of the absorbent liquor, no appreciable change in composition being assumed to occur. A bell suspended on a balance beam and actuating a pointer on a scale is caused to drop to the level of the liquid surface by the closing of the gas inlet valve. The amount of sinking of the bell, which has thus entrapped a gas sample at atmospheric pressure, consequent on the absorption of carbon dioxide, is recorded. The apparatus may be operated mechanically or by hand. C. Irwin.

Fire-extinguishing liquids. Knock-out Fire Extinguishers, Ltd. From Soc. Bouillon Frères (E.P. 260,535, 13.9.26).—A substance which prevents the formation of phosgene (*e.g.*, an aromatic hydrocarbon) and a fatty substance are added to a fire-extinguishing liquid which normally evolves phosgene, such as carbon tetrachloride, tetrachloroethylene, or tetrachloroethane.

H. Holmes.

Lubricating compound. T. S. Hamilton (U.S.P. 1,599,963, 14.9.26. Appl., 8.8.25).—A mixture of cellulose ester and graphite. H. Holmes.

Crushing machines (E.P. 258,225).—See II.

Heat exchange apparatus (E.P. 260,066).—See II.

Atomising and desiccating liquids (E.P. 256,190). **Apparatus for drying liquids** (E.P. 260,453).—See XIX.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

The initial period in coal dust explosions. H. Sano (J. Fac. Eng. Tokyo, 1926, **16**, 195—236).—Experiments on a laboratory scale have been made to determine the factors governing the ignition and the propagation of flame in coal dust–air mixtures for a wide range of Japanese coals. The ignition temperature of a mixture of coal dust and air is considerably influenced by the area of the igniting surface, and the time of contact therewith, whilst the ignition of powdered charcoal, which evolves no inflammable volatile matter on heating, is uninfluenced by these factors. The combustion of coal dust mixtures is preceded by a partial distillation of the coal, until the setting up of the explosion wave, the velocity of which is too great for such pre-distillation to occur. When the concentration of the coal dust is sufficiently high that the evolved volatile products themselves form a combustible mixture with the air, combustion proceeds by the preferential burning of this gaseous mixture. Using the same area of igniting surface, the ignition temperature varies little for different Japanese coals, but the length of flame produced from the same amounts of coal dust increases approximately inversely as the ratio (fixed carbon + ash)/(volatile matter). For a point source of heat, the inflammability of coal dust–air mixtures is approximately proportional to the volatile matter content of the coal, but more nearly proportional to the amount of matter extractable by phenol. The minimum concentration of coal dust (through a 200-mesh sieve) necessary for continued propagation of flame averages about 80 g./cub. m., varying inversely as the volatile matter content for different coals. The relative critical temperatures necessary to start combustion in such mixtures do not differ much from one coal to another (1000—1100°), and the pressures developed are of the same order of magnitude. The upper limit of concentration necessary for continued propagation of flame is about 1000 g./cub. m., the maximum pressure being developed by mixtures containing about 300 g./cub. m. The inflammability of the mixtures is not markedly influenced by the presence of less than 2% of methane in the air; the danger of explosion is, however, greatly increased by the presence of more than that amount. The presence of an inorganic dust markedly inhibits the combustion. The fineness of the coal dust has a considerable influence on the inflammability of the mixtures; dust finer than 40—60 mesh is necessary to form an explosive mixture, the danger of explosion thereafter increasing with increasing fineness. The danger of explosion is less with an oxidised than with a freshly mined coal. The lower limit of concentration of coal dust necessary for combustion is lowered by a rise in the initial temperature of the mixture, but is not markedly influenced by an increased pressure. The results are discussed from the point of view of mining practice. A. B. Manning.

Resin inclusions in bituminous coals. R. Wiggington (Fuel, 1926, **5**, 476—478).—Thirteen samples of resin inclusions from bituminous coals were examined. The resin exists as transparent red-brown flakes, soluble in chloroform; they do not contain nitrogen and sulphur, and have a higher carbon and hydrogen content than the accompanying coal. Fractionation with alkaline solvents was unsuccessful, but the resin was separated into two fractions by means of alcohol, and these two fractions were further subdivided by ethyl ether, the insoluble fraction being further treated with benzene and the soluble fraction with light petroleum. The presence of hydroxyl groups was detected in all the fractions by acetylation. Hydrocarbons were either present in small quantities or entirely absent. The mean molecular weight of the fractions was of the order of 1000. The chloroform extracts of bituminous coals closely resemble the resins in general appearance, ultimate analysis, and behaviour on heating, but differ in the subsequent fractionations; the resins, therefore, do not represent normal constituents of bituminous coals.
A. C. Monkhouse.

Modification of Brunck's method of determining sulphur in solid fuels. V. Schön and F. Vykypiël (Chem.-Ztg., 1926, **50**, 673—674).—A mixture of 1 g. of the finely-powdered fuel with 1 g. of a mixture containing 10% of cobaltic oxide, 60% of magnesia, and 30% of sodium carbonate is heated in a porcelain boat in a glass tube through which a current of oxygen is passed. As soon as the reaction commences, the heat is reduced somewhat, and the gas stream continued for 10—20 min. after action has ceased. The issuing gases are passed through ammoniacal hydrogen peroxide to collect any trace of sulphur dioxide formed. The residue in the boat is leached with hot water, and the sulphate determined as usual in the Eschka method. The processs is also applicable to the analysis of pyrites using 0·2 g. of the mineral to 2 g. of the catalyst mixture.
A. R. Powell.

Report of test on the "Fusion" rotary retort at the works of Electro-Bleach and By-Products, Ltd., Cledford, Cheshire. C. H. Lander (Dept. Sci. Ind. Res., 1926, 21 pp.).—The retort consists of a horizontally rotating drum, with a rated capacity of 5 tons per day, and may contain "breakers" intended to churn up the charge passing through the retort under its own head. It is fitted with a charging hopper, a discharging device, a "dust-catcher" for the gas, which then passes through an exhauster and an oil scrubber to a holder, and tar and liquor tanks. The retort is heated by the hot products of combustion from a furnace burning blast-furnace coke. The temperature of the charge during the test probably did not exceed 550°. Welbeck cannel coal was used with a small admixture of bituminous coal. A throughput of nearly 3½ tons per day was maintained. The yields of products per ton of coal were: coke, 11·6 cwt.; tar and oils, 50·23 gal.; crude spirit from gas scrubbing, 3·75 gal.; refined spirit from tar, 2·7 gal.; gas, 2740 cub. ft. of 1070 B.Th.U./cub. ft. (gross) = 29·3 therms. R. A. A. Taylor.

Influence of coking conditions and the addition [of inorganic oxides] to the coal on the properties of coke. H. Bähr and F. Fallböhmer (Gas- u. Wasserfach, 1926, **69**, 909—912, 929—932, 943—947; cf. B., 1925, 159).—Determinations have been made of the ignition temperature (Bunte and Kölmel, B., 1923, 132A), reaction temperature (Fischer, Breuer, and Broche, B., 1923, 256A), reactivity (Bähr, B., 1924, 160), specific gravity, porosity, and hardness of cokes made by

carbonising coal at 550°, 750°, 950°, and 1150° respectively. A Westphalian coal, the ash in which had been reduced to 0·7—0·8%, was carbonised, either alone or with the addition of small amounts of ferric oxide, silica, or other inorganic oxides, in a specially-designed electrically-heated retort of ½ kg. capacity. The ignition temperature, reaction temperature, and reactivity were determined successively on the same sample of coke. The ignition temperatures and reaction temperatures rise with the temperature of carbonisation, but are unaffected by the addition of inorganic oxides to the coal. The reactivity, determined by the carbon monoxide produced when oxygen, air, or carbon dioxide is passed over the coke at 950°, falls from 32·2% for the 550° coke to 11·5% for the 1150° coke. Addition of silica does not appreciably affect the reactivity, but addition of ferric oxide, manganese dioxide, or copper oxide causes a marked increase, 3% of ferric oxide, for example, raising the values to 60·4% for the 550° coke and to 48·2% for the 1150° coke. In cokes prepared at 1150° this catalytic effect of the ferric oxide is annulled by the addition of an equal amount of silica, due to the formation of the non-active ferrous silicate. Subsequent heating for some hours at 1150° of the cokes made at the lower temperatures does not affect their reactivity unless the rate of heating is too rapid, when small changes occur; the reactivity, therefore, is independent of the volatile matter content. Graphitising the cokes by passing benzene vapour over them at 1150° reduces the reactivities of all of them to the normal value for the 1150° coke. Reactivity determinations made at 1100° give the same relative values as those made at 950°. The true specific gravity increases with the temperature of carbonisation, but neither the apparent specific gravity nor the porosity exhibits any marked regularities. The resistance to abrasion in general increases with rise of temperature of carbonisation, and with the addition of inorganic oxides. It is concluded that the modification of carbon present in the coke has the greatest influence on its pyrochemical and physical properties, the modification of carbon being a function of the carbonisation temperature. The reactivity is influenced in particular by the addition of certain catalytically-acting oxides. In the absence of such catalysts the ignition temperatures, reaction temperatures, or reactivities are of equal value in judging the pyrochemical properties of cokes. A. B. MANNING.

Carbonyl number of coals and its relation to the age [of the coal deposit] and the degree of weathering. H. STRACHE and A. BRANDL (Brennstoff-Chem., 1926, **7**, 341—344).—The carbonyl oxygen has been determined in a number of coals ranging from lignites to anthracites. 0·15—0·2 g. of the sample was warmed with a solution of phenylhydrazine, and, after filtration, excess of the latter determined by oxidation with Fehling's solution and measurement of the nitrogen evolved. The results, expressed as percentage of carbonyl oxygen in the dry and ash-free coal, varied from about 1% for anthracites to between 2 and 3% for bituminous coals, and between 3 and 4% for brown coals and lignites. Lignin gave relatively little carbonyl oxygen, and cellulose none. Weathering of the coal was generally accompanied by a marked decrease in the carbonyl number. A. B. MANNING.

Purification of effluents from by-product plants of coke ovens and gas works. H. BACH (Gas- u. Wasserfach, 1926, **69**, 912—915, 932—935, 947—952).—An account is given of the problems arising in the disposal of gas works' and coke oven effluents, and the methods that have been proposed for treating such effluents. The most successful is that depending on the oxidation of the impurities (phenol, thiocyanates, etc.) by bacterial action (cf. Fowler, Ardern, and Lockett, J.S.C.I., 1911, **30**, 174). The Emscher filter, a plant designed by the author and applying this method, consists of a contact tank filled with clinker which is inoculated by a preliminary treatment with sewage. The diluted effluent passes down through the tank, while at the same time a current of air is blown up through it (cf. B., 1926, 646). An effluent of medium phenol content requires dilution with about four times its volume of the purified effluent, containing preferably some liquid sewage. The necessary conditions for the successful operation of the plant are (*a*) the continued presence of the active bacteria, (*b*) sufficient dilution of the effluent, (*c*) the effluent must be alkaline, (*d*) suitable temperature regulation, (*e*) abundant air supply, and (*f*) the addition of a certain amount of nutriment for the bacteria. The construction and operation of a plant in use at Altenessen are described in detail. This plant reduces the phenol content of a coke oven by-product effluent from 1300—2100 to 15—40 pts./million, the thiocyanate content from 100—160 to 0—6, and the sulphur content from 300—450 to 10—20 pts./million; it deals with about 1 kg. of phenol per cub. m. of contact material per 24 hrs.

A. B. MANNING.

Path of travel of the gases in the coke oven. W. E. DAVIES (Gas World, 1926, **85**, 128—133).—From a survey of the work of various investigators it is concluded that after the first rapid coking of the coal adjoining the walls there is a change from a flow of gas towards the centre of the oven to a dual flow in an outward and inward direction, the former increasing as the plastic layer moves inwards and as fissures are formed in the coke. The plastic layer acts as a partition wall, and two pressure-gradients are formed, one on either side of it. When the two plastic layers meet, a local high pressure results, and an outward flow occurs until the coke fissures in the centre. Emphasis is laid on the size of coal carbonised, the plastic resistance decreasing as the size of coal particle is increased. A narrow oven is superior to the wide oven, as it prevents too large a ratio of outward travel of the gases. A. C. MONKHOUSE.

General formula for calculating the calorific value of solid fossil fuels from their ultimate analyses. W. STEUER (Brennstoff-Chem., 1926, **7**, 344—347).—The proposed formula is based on the assumption that the oxygen may be divided equally between the carbon and the hydrogen, that the combined oxygen and hydrogen contribute nothing to the calorific value, whilst the combined oxygen and carbon contribute an amount equal to the heat of combustion of the equivalent carbon monoxide. Thus the gross calorific value is given by $H = 81(C - 3O/8) + 57(3O/8) + 345(H - O/16) + 25S$, from which $6(W + 9H)$ must be subtracted to give the net value. The formula is applicable over the whole range of fuels from peat to bituminous

coal, the maximum differences between the calculated and observed calorific values being about 2%.

A. B. MANNING.

Catalytic cracking of heavy fractions of petroleum. C. MAZZETTI (Annali Chim. Appl., 1926, **16**, 414—429).—American fuel oil, *d* 0·885, calorific value 10·8 kg.-cal., b.p. (main fractions) 200—300°, is cracked by passage through an iron tube heated in an electric furnace at 500—800°. The proportion of low-boiling products increases as the temperature of cracking rises, reaching a maximum at about 700°. The results using (*a*) an empty tube; (*b*) a tube containing carbon formed in the cracking process; and (*c*) a tube filled with gas coke, are compared: the yield of gas is in the order $a < b < c$, with a maximum of 750° for *c*; the gas from *c* contains the larger proportion of unsaturated products, which decrease as the temperature rises, but have an optimum temperature in case *b*.

E. W. WIGNALL.

Isolation and identification of certain thiophen compounds occurring in shale-oil. H. SCHEIBLER (Z. angew. Chem., 1926, **39**, 1397—1398).—Bavarian and Tyrol shale-oils have a high sulphur content and are highly unsaturated, so that they are incapable of being refined by ordinary processes. An "ichthyol crude-oil," for example, was found to contain 50% of thiophen compounds. Such an oil is treated with concentrated sulphuric acid and then neutralised with ammonia, yielding ichthyol, which forms an aqueous emulsion in which the thiophen compounds are still present. A similar result is obtained in the case of purification with soda-lime and sodamide. In order to isolate the thiophens the purified oil was fractionated and treated with acetyl chloride and aluminium chloride in quantity equivalent to the sulphur content calculated as thiophen. The ketones so obtained were converted into *p*-nitrophenylhydrazones and fractionally crystallised. In this way 2-*n*-butylthiophen, 3-*n*-propylthiophen and 2-*iso*propylthiophen were identified. The Friedel-Krafts reaction is applicable to all thiophen compounds containing one or more hydrogen atoms in the ring, and is much superior to the mercuric chloride method.

C. IRWIN.

Examination of lubricating oil [after use in automobile engines]. P. V. ROSEWARNE (Canad. Chem. Met., 1926, **10**, 233—236).—The changes in lubricating oils used in 17 cars were followed. It was found that the dilution on use varied from 0·5 to 20%. The diluent was found to coincide closely in physical properties with the first 30% distilled from commercial kerosene. After removal of the diluent the residue was in general found to be more viscous than the original lubricating oil; this is thought to be due to oxidation. The carbon residue value of an oil was found to increase considerably with use. The lowering of flash point of an oil may be taken as a rough estimate of the percentage of diluent present in it.

W. N. HOYTE.

Spontaneous ignition temperatures of inflammable liquids and the effects of anti-knock materials. Y. TANAKA and Y. NAGAI (J. Soc. Chem. Ind. Japan, 1926, **29**, 266—272).—The spontaneous ignition temperatures of various inflammable liquids in a current of oxygen were determined, using an improved form of Moore's apparatus, as follows: Aero-gasoline (b.p. 35—118°, main fraction 40—100°), 310°; kerosene (b.p. 104—294°, main fraction 140—280°), 267°; *n*-hexane (b.p. 68·5—68·7°), 285°; methyl*cyclo*pentane (b.p. 72—72·2°), 329°; *cyclo*hexane (b.p. 80·8°), 318°; *iso*heptane (impure, b.p. 90·5—90·9°), 290°; *n*-heptane (impure, b.p. 96·5—97°), 298°; methyl*cyclo*hexane (b.p. 100·75—101°), 312°; ethyl ether (b.p. 34·6°), 200°; ethyl ether (containing 0·8% of water), 340°; methyl alcohol (pure), >513°; *n*-propyl alcohol (b.p. 96—97°), 328°; *n*-butyl alcohol (b.p. 117·6—117·9°), 334°; *iso*butyl alcohol (b.p. 107·1—107·6°), 364°; amyl alcohol (b.p. 129·5—130·2°), 332°; fusel oil (dehydrated), 322°; acetone (pure), >504°; benzene (pure), >513°. The presence of very small amounts of ethyl selenide or lead tetraethyl caused a great rise in the ignition temperatures of alcohols, but little or none with other inflammable liquids.

K. KASHIMA.

Effects of aromatic amines and pyridine on the spontaneous ignition temperatures of inflammable liquids. Y. TANAKA and Y. NAGAI (J. Soc. Chem. Ind. Japan, 1926, **29**, 272—274; cf. preceding abstract).—The addition of aniline, *m*- and *p*-toluidines, *m*-4-xylidine, and pyridine raised the spontaneous ignition temperature of ethyl alcohol, but had little effect on methyl*cyclo*hexane and ether.

K. KASHIMA.

PATENTS.

Reducing or pulverising coal and other materials. H. G. LYKKEN (E.P. 258,109, 28.10.25).—The coal passes from a hopper through a crushing roller regulated by a plate into a chamber containing a shaft with arms rotating at high speed. The coal is thrown against the sides of the chamber and disintegrated. Air is drawn in by means of an exhauster, and the particles, after meeting bars arranged as baffles across the chamber, pass up through a series of vanes so arranged as to give a whirling motion to the particles. The heavier particles fall back into a cone-shaped chamber and thence, by a cone discharge, are returned to the main chamber. The fine particles are drawn upwards to the exhauster fan and conveyed to the combustion chamber; the fineness of the coal particles is controlled by the air admission. Arrangements are made for the withdrawal of heavy particles from the bottom of the chamber and for a safety release in the preliminary crusher.

A. C. MONKHOUSE.

Process and apparatus for low-temperature distillation of coal. M. W. TRAVERS and F. W. CLARK (E.P. 259,295, 6.7.26).—Coal is carbonised in a chamber at a temperature preferably below 600° by the admission of highly superheated steam into one or other of two chequered regenerators, which are alternately heated by the combustion of producer gas and air. Saturated steam is also introduced into the coke chamber, where it abstracts heat from the coke and passes superheated into the carbonising zone. The distillation products together with superheated steam pass into a scrubber, fed with hot water, whereby the quantity of steam carried by the distillation gases is increased and the degree of supersaturation decreased. A thermo-compressor of the jet type, fed with live steam, draws the steam and distillation products from the scrubber and compresses them into a heat exchanger, where steam is raised out of

contact with the distillation products. Steam raised in the boiler passes to both the superheaters and the coke chamber. Condensed water and tar flow into an economiser, where their heat is transferred to the water feeding the boiler and the scrubber. Uncondensed gases are cooled in a condenser. Coal distilled by this system yields the maximum amount of volatile products, but the minimum of gas. S. Pexton.

Manufacture of coke. Soc. Anon. les Agglomérés du Brabant, and A. Reifferscheidt (E.P. 259,427. 21.12.25).—Anthracite, coke, or lignite dust is triturated cold with a mixture of coal tar pitch and water in the proportions of 100 pts. of dust, 20 pts. of pitch, and 12 pts. of water, and the mixture is heated in retorts to 900—1000°. During the distillation a compound, $C_{22}H_{14}O_5$, is formed, which acts as a coking agent. S. Pexton.

Coke ovens. Semet-Solvay Co., Assees. of C. H. Hughes (E.P. 253,876, 18.5.26. Conv., 19.6.25).—Coke ovens with horizontal flues are each provided with a pair of regenerators situated immediately below the oven sole. Each regenerator communicates directly at its own end with the flues on both sides of the oven, there being no intermediate sole flue. In this way the air, preheated in one or other regenerator, passes direct to the combustion flues. The upper parts of the regenerators supply sufficient heat to carbonise the coal at the sole of the oven. S. Pexton.

Crushing machines [for coal, coke, etc.]. A. Lozai and A. Lerciu (E.P. 258,225, 29.6.26. Conv., 11.9.25).—A crushing machine for comparatively soft materials such as coal comprises a number of groups of picks which are reciprocated vertically by a crank-shaft or other means. The material is conveyed under the picks on a spring-mounted table, which is connected to the driving gear so that when an uncrushable substance, such as a lump of metal, gets under an earlier group of picks the table will be depressed and stop the machine, which restarts when the obstruction is removed. B. M. Venables.

Draining or straining coal and other slimes. Simon-Carves, Ltd., and A. Robinson (E.P. 260,329, 20.7.25).—Coal or mineral slurry is drained on an inclined sieve, freely suspended, and given a combined reciprocating and vibrating motion by means of a rotating cam member. It may then be passed over a horizontal vibrating sieve. C. Irwin.

Manufacture of coal gas. H. J. Toogood, and R. Dempster & Sons, Ltd. (E.P. 260,398, 22.9.25).—A setting of gas retorts communicates by valves with descension pipes down which liquor is sprayed, and which lead to seal pots and a foul-main below the level of the charging and discharging floors. The gas is thereby rapidly cooled, partially washed, and the naphthalene content reduced. A. B. Manning.

Coke oven and the like. A. Roberts (U.S.P. 1,601,741, 5.10.26. Appl., 13.9.20).—A design for a coke oven heating wall of silica. A. B. Manning.

Treatment of oil shales. M. J. Trumble (U.S.P. 1,598,831, 7.9.26. Appl., 15.5.22).—A mixture of steam and hydrogen produced by the decomposition of steam is caused to traverse a charge of oil shale contained in an externally-heated metal retort. The vapours produced are withdrawn and condensed. S. Pexton.

Destructive distillation of solid bituminous materials. A. M. Smith (U.S.P. 1,602,128, 5.10.26. Appl., 21.12.22).—The apparatus consists of a vertical retort, a condensing system, a gas scrubber, and a gas heater, so arranged that the material is distilled by the re-circulation through the retort of the heated gaseous distillation products. A. B. Manning.

Distillation of easily-decomposable substances. W. K. Lewis, Assr. to Humble Oil & Refining Co. (U.S.P. 1,599,824—5, 14.9.26. Appl., [a] 10.11.22; [b] 30.12.22).—The liquid to be distilled is heated under diminished pressure in a still, and the vapours pass up an empty column, the sides of which are jacketed with a hot liquid. (a) A stack of pipes at the top of the column cooled by an internal current of liquid, or (b) an outwardly cooled, inclined pipe partially condenses the still vapours. The run-back is caught by a pan situated beneath the cooling pipe, and is separately collected. The uncondensed vapours pass to the main condenser. W. N. Hoyte.

Uniform revivification of activated carbon. V. S. Allien, Assr. to Darco Sales Corp. (U.S.P. 1,599,072, 7.9.26. Appl., 16.7.24).—Thin layers of the carbon are placed on a series of flat shelves arranged in tiers in a chamber, which is heated internally so that the carbon is out of contact with the heating gases. There is a gradual increase in temperature from the top to the bottom of the chamber, and means are provided for moving the carbon along the shelves. A. C. Monkhouse.

Producing enriched water-gas. W. E. Trent, Assr. to Trent Process Corp. (U.S.P. 1,600,375, 21.9.26. Appl., 21.6.21).—During the period of gas making in a water-gas plant, a fuel of plastic consistency containing oil, coal, and water, is fed into the generator in a ribbon-like form; the volatile portions enrich the gas and the residual carbon is deposited on the fuel bed. A. C. Monkhouse.

Converting [cracking] hydrocarbons. R. T. Pollock, Assr. to Universal Oil Products Co. (U.S.P. 1,602,990, 12.10.26. Appl., 25.11.19).—Oil is cracked in a pipe still, at 400°, under a pressure of about 9 atm., and the products are led through an expanding chamber into a dephlegmator, in which they are cooled and freed from high-boiling substances by treatment with fresh, uncracked oil. Some of the mixture of oil and condensate is passed to the still, and a portion of the remainder is circulated through the dephlegmator, fresh oil being added as required. T. S. Wheeler.

Treating [cracking] hydrocarbon oils. J. B. Weaver, Assr. to Oil Products Co. (U.S.P. 1,601,786, 5.10.26. Appl., 15.6.25).—Oil is passed through a pipe still, in which it is heated to 370°, at about 2 atm., into an expander, where heavy residues are removed, and a converter, in which it is mixed with steam and led at about 600° over ferric oxide. The oil cracks, and the unsaturated products take up hydrogen from the steam. The issuing vapours are rapidly cooled to 235° by treatment with fresh, uncracked oil, at which temperature

most of the carbon present separates. The fresh oil and the high-boiling constituents of the cracked products are passed to the still. T. S. WHEELER.

Treatment of [hydrocarbon] oils. E. O. LINTON (U.S.P. 1,599,777, 14.9.26. Appl., 8.12.20).—In order to free an oil from asphalt, it is fed continuously to a still where the heavy oils are vaporised; it then flows to the open still chamber where, in its flow over a wall maintained at a temperature of 460°, the vaporisable constituents are removed. All vapours are condensed, and the asphalt is removed from the bottom of the still. W. N. HOYTE.

Purification of [hydrocarbon] oils. SHARPLES SPECIALTY Co., Assees. to L. H. CLARK (E.P. 243,666, 21.10.25. Conv., 29.11.24).—In order to purify such oils as have been used in switch gear, contact breakers, and internal-combustion engines, the oil is beaten up into an emulsion with dilute solutions of substances such as sodium hydroxide, phosphate, or silicate. The emulsion is subsequently broken by a centrifuge and, if necessary, a clarifier. Further purification can be effected by the addition of fuller's earth to the emulsion. In general it is found that colloidal carbon and dirt are removed in the aqueous layer, and that the resulting oil is more resistant to emulsification than before. W. N. HOYTE.

Purification of [hydrocarbon] oils. P. W. PRUTZMAN and P. D. BARTON, Assrs. to GENERAL PETROLEUM CORP. (U.S.P. 1,599,715, 14.9.26. Appl., 25.6.25).—An emulsion is produced by mixing the oil with a wet absorbent material, with oil as the continuous phase. By agitation with steam the emulsion is changed to one in which water is the continuous phase. W. N. HOYTE.

Oil-treating composition. H. REINBOLD (U.S.P. 1,600,845, 21.9.26. Appl., 15.8.25).—A hydrous aluminosilicate, *e.g.*, bentonite, is intimately mixed with 25—40% of dry sodium hydroxide, and, when reaction is completed, the mass is treated with chlorine. The product is an intimate mixture of a colloidal hydrous sodium aluminosilicate and sodium hypochlorite, and can be applied to bleaching and desulphurising oils. It is stable and free from acid. T. S. WHEELER.

Heat exchange apparatus [for lubricating oil]. E. STORONI (E.P. 260,066, 12.8.25).—The oil to be cooled flows between the walls of pairs of concentric tubes, and cooling water circulates in contact with the other surfaces of these walls. Conduits with separate valves are provided for the water passing through the inner tubes and that circulating on the outside of the outer tubes, thus enabling a single or a double circulation to be effected, according to the amount of oil to be cooled. The difference between the outer radius of the inner tube and the inner radius of the outer tube of each pair is from 0·5 to 2·0 mm., corresponding to about twice the thickness of the layer of liquid adhering to the walls. The ends of the tubes are fixed by screw glands to header plates. H. HOLMES.

Refining mineral lubricating oils. J. W. WEIR (U.S.P. 1,603,174, 12.10.26. Appl., 14.5.25).—Oil is treated with sulphuric acid and, after removing the greater part of the sludge formed on settling, is agitated with a fresh pulverised adsorbent material at a temperature below which the sludge decomposes. The oil is separated from the adsorbent and deleterious matter, and, after treatment with further adsorbent, is heated sufficiently to decompose the remainder of the sludge, with liberation of sulphur dioxide, and again separated from the oil. H. ROYAL-DAWSON.

Oil-refining still. F. C. MOORE and P. VANDERVORT (U.S.P. 1,599,833, 14.9.26. Appl., 4.9.24).—The oil is heated in a pipe still. A long firebox extends underneath the entire still, and, by a suitable arrangement of baffles, the hot gases pass by a tortuous path over the stack of pipes. W. N. HOYTE.

Dehydrating crude petroleum oil. H. O. BALLARD (U.S.P. 1,600,030, 14.9.26. Appl., 10.4.19).—The oil is heated and passed in a continuous stream through a closed chamber; the emulsion which settles out there is removed from time to time. W. N. HOYTE.

Intermingling of liquids [motor fuels] in any desired proportion. H. KAUWERTZ (E.P. 258,890, 24.9.26. Conv., 28.9.25).

Sectional retort (U.S.P. 1,602,678). See I.

III.—TAR AND TAR PRODUCTS.

Complex aromatic hydrocarbons in low-temperature tar. G. T. MORGAN and D. D. PRATT (Nature, 1926, **118**, 805).—The fraction, b.p. 313—360°, of a tar produced by distillation of Pooley Hall–Wearmouth coal, carbonised at 600°, yielded a complex mixture of aromatic hydrocarbons, β-methylanthracene predominating. A. A. ELDRIDGE.

Tar distillate washes. GOODWIN and others.—See XVI.

PATENTS.

Treatment of spent acids obtained in refining hydrocarbons. DORMAN, LONG & Co., LTD., J. A. ROELOFSEN, and L. SHUTTLEWORTH (E.P. 260,514, 12.6.26).—Acid tar from the washing of crude benzol etc. is first steamed to remove hydrocarbons, and is then diluted with 1—3 times its volume of ammonium sulphate mother-liquor. The resins are precipitated and skimmed off, and the acid liquor, after filtration with sand, if desired, is suitable for the manufacture of further ammonium sulphate. C. IRWIN.

Obtaining phenols from ammoniacal liquor or industrial waste liquors. ZECHE M. STINNES (E.P. 249,111, 5.3.26. Conv., 13.3.25).—The liquors are treated with organic bases, such as pyridine, quinoline, etc., or with tar oils from which the phenols have been removed, either alone or as a mixture with benzene or its homologues. Ammonia liquor, washed with 20% by volume of a mixture of 80% of benzene and 20% of organic bases of b.p. 230°, completely removed the phenols as compared with a 65% removal using benzene alone. A. C. MONKHOUSE.

IV.—DYESTUFFS AND INTERMEDIATES.

Determination of aniline, especially in dilute solutions. A. V. PAMFILOV (Z. anal. Chem., 1926, **69**, 282—292).—Comparative tests on the determination of aniline in dilute solutions by the diazotising, acidimetric, colorimetric, and bromometric methods indicate that the

last gives the most nearly correct results. The determination is best carried out by direct titration of the nearly neutral solution with a solution of bromine in N-potassium bromide using indigo-carmine as indicator. The indirect method of adding an excess of bromate to the acid aniline solution and determining the excess iodometrically is satisfactory if sufficient time is allowed to elapse before titration to ensure complete bromination of the aniline. A. R. Powell.

Adsorptive properties of an active charcoal [for dyes]. Mazzetti.—See II.

Patents.

Manufacture of intermediates and [azo] dyestuffs. Brit. Dyestuffs Corp., Ltd., K. H. Saunders, and M. Mendoza (E.P. 260,058, 31.7.25).—5-Thiolsalicylic acid (cf. Stewart, J.C.S., 1922, **121**, 2560) or its sodium salt, its homologues, or substitution products are condensed with aromatic nitro-compounds containing a labile nuclear halogen atom, yielding nitro-derivatives of 3-carboxy-4-hydroxydiphenyl sulphide, which are then reduced to the corresponding amino-compounds. *E.g.*, 10 pts. of 5-thiolsalicylic acid, 15·85 pts. of sodium 4-chloro-3-nitrobenzenesulphonate, and 12 pts. of sodium acetate are boiled together in aqueous solution under reflux for some hours, then cooled and acidified, when the product is precipitated. The nitro-compound so formed is reduced to the amino-compound by one of the usual methods. The monoamino-compounds obtained by such methods can be diazotised and used as first components for mono- and poly-azo dyes. Some of the *m*-diamino-compounds may be used as second components. The diamino-compounds may also be partly acylated, and the free amino-group diazotised and coupled with a second component. The new dyes can be fixed on animal or vegetable fibres by metallic mordants with little or no change in shade, and after fixation are fast to soap, milling, and potting. From 5-chlorosulphonyl-2-hydroxy-3-toluic acid (G.P. 264,786; B., 1913, 1003) by reduction below 0° with zinc dust and alcohol, is obtained 5-sulphino-2-hydroxy-3-toluic acid, from which, on reduction at ordinary temperature with zinc dust and mineral acid, 5-*thiol*-2-*hydroxy*-3-*toluic acid*, m.p. 180—182°, is formed. 3-Chlorosalicylic acid, on treatment with chlorosulphonic acid, gives 3-chloro-5-chlorosulphonylsalicylic acid, which is reduced through the sulphinic acid to 3 : 3′-*dichloro*-4 : 4′-*dihydroxy*-5 : 5′-*dicarboxydiphenyl disulphide*, m.p. 258—259°. The disulphide is then further reduced in caustic alkaline solution by sodium hyposulphite to 5-*thiol*-3-*chlorosalicylic acid*, m.p. 210°. A. Davidson.

Sulphur black and similar sulphur dyes. M. Paley (U.S.P. 1,598,303, 31.8.26. Appl., 19.5.23. Renewed 27.1.26).—Sodium dinitrophenoxide is thionated with a polysulphide obtained by mixing 69·2% of sodium sulphide ($Na_2S_{4\cdot5}$), 27·7% of flowers of sulphur, and 3·1% of crude sulphur. This process permits of an increased yield of 20% of sulphur black of greater tinctorial power than is usually obtained.
R. B. Clarke.

Process for reducing nitro-compounds. W. Carpmael. From I.G. Farbenind. A.-G. (E.P. 260,186, 10.6.26).—A mixture of the vapour of the nitro-compound with excess of hydrogen is passed over silica gel impregnated with a small quantity of a heavy metal such as copper, iron, or nickel, or their salts or oxides. *E.g.*, a mixture of nitrobenzene vapour and hydrogen is passed at about 200° through granular silica gel on which a little copper hydroxide has been precipitated. Under these conditions the copper oxide formed is reduced to metallic copper. A very good yield of aniline is obtained. For the reduction of the nitrotoluenes the temperature should be 220—225° and for α-nitronaphthalene 240°. When the nitro-compound boils at a high temperature the process may be carried out under reduced pressure. 3-*Aminopyridine*, m.p. 63°, is obtained by this process from 3-nitropyridine. Nitromethane is converted at 180° into methylamine. A. Davidson.

Nitriles of the perylene series (E.P. 254,310).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of α-cellulose. P. Waentig (Papier-Fabr., 1926, **24**, 689—690).—By carefully standardising the method of washing, or by first washing the cellulose with fresh 8% sodium hydroxide solution and then with water (cf. B., 1922, 935 A), erroneous results owing to the unavoidable dilution of the solution and the consequent reprecipitation of some soluble cellulose are avoided. Celluloses such as those normally used in the artificial silk industry can be dried for analysis at a temperature not exceeding 110° without appreciably affecting the α-cellulose content, but it is preferable to carry out the determination on the air-dry material and correct for the moisture present. The sample should not be disintegrated mechanically, as this causes a reduction in the α-cellulose content. The result is also influenced by the time and temperature of the immersion, and by the ratio of the volume of sodium hydroxide solution to the weight of cellulose. These factors should, therefore, be rigidly controlled.
W. J. Powell.

Determination of cellulose and mechanical pulp in paper. O. M. Halse (Papir-J., 1926, **10**, 121; Papier-Fabr., 1926, **24**, 631).—1 g. of the paper is shaken with 50 c.c. of 38% hydrochloric acid, 5 c.c. of concentrated sulphuric acid being added after the paper has been thoroughly soaked. The solution is shaken and allowed to remain overnight, then diluted to 500 c.c. and boiled. The lignin is collected in a porous crucible, washed, and dried at 100°. The calculation is made on the lignin values for 1 g. of mechanical pulp, 0·266 g.; and for 1 g. of sulphite-cellulose, 0·030 g. If the paper contains loading, this is determined by ignition, and a correction is made on the assumption that 88 pts. of ash are equivalent to 100 pts. of air-dry loading. J. F. Briggs.

Bleaching of sulphite-cellulose. Rys.—See VI.

Cellulose sausage casings. Henderson and Dietrich.—See XIX.

Patents.

Moth-proofing composition [for woollen fabrics]. Larvex Corp., Assees. of M. G. Minaeff (E.P. 236,218. 24.6.24. Conv., 24.6.24).—Woollen materials are treated with a solution containing a toxic substance and sub-

stances which promote rapid and thorough penetration of the materials by the solution. Caprylic acid and higher aliphatic acids and their salts are satisfactory wetting-out agents, and are used together with a protective colloid such as gelatin. The presence of a free organic acid, *e.g.*, citric acid, improves the wetting-out power of the composition and the permanence of the proofing treatment. A. J. Hall.

Removal of stains from fabrics. A. H. R. S. Poole (E.P. 259,396, 30.10.25).—A composition which removes stains without injuring the fabrics consists of 1 part of ammonium carbonate, $\frac{3}{4}$ part of potassium nitrate, $\frac{1}{2}$ part of flaked soap, and 20 parts of water. The addition of a small quantity of sodium chloride prevents the composition affecting dyes.
F. G. Crosse.

Cement for fabrics. N. C. Amen (E.P. 258,698 9.7.25).—A liquid waterproof cement suitable for repairing canvas consists of nitrocellulose, benzene, and acetone, to which may be added a small proportion of amyl or ethyl acetate for the purpose of preventing "blushing." A. J. Hall.

Producing halfstuffs and cellulose from vegetable fibres [peat]. R. Runkel (U.S.P. 1,602,253, 5.10.26. Appl., 31.5.23. Conv., 31.7.22).—Peat, saturated with water, is subjected to sudden heating, or to cooling below 0°, and is then treated three times, alternately, for $\frac{3}{4}$-hr., with concentrated sodium hydroxide and chlorine. Halfstuff, suitable for the manufacture of paper pulp and pasteboard, is thus obtained. By continuing the treatment, cellulose adapted to the manufacture of paper is produced. T. S. Wheeler.

Varnishing leather [with cellulose acetate] (E.P. 255,803).—See XV.

Lubricating compound (U.S.P. 1,599,963).—See I.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching of sulphite-cellulose: influence of chlorates. L. Rys (Papier-Fabr., 1926, 24, 625—627).—Contrary to the statement of Schwalbe and Wenzl (B., 1923, 50 A), it is now proved that the chlorates present in bleach liquor take no part in the reaction, and remain practically unchanged in amount, both with warm and cold bleaching. The addition of chlorate to the bleach liquor has no effect on the chemical equilibrium of the bleaching process, the physical conditions of the operation are unchanged, and the chemical constants of the bleached pulp are unaffected.
J. F. Briggs.

Absorption of acid and basic dyes by cationic and anionic chrome-tanned hide powder. Gustavson.—See XV.

Patents.

Fast dyeings on wool. J. Y. Johnson. From Badische Anilin u. Soda Fabr. (E.P. 260,339, 27.7.25).—A large variety of shades having excellent fastness are obtained on wool by impregnating this fibre with a sulphonated azo dye (preferably derived from naphthalene) containing suitable hydroxy- and/or amino-groups, and then treating it with a diazo compound, the dye and the diazo compound being chosen so that the resulting tetrazo compound (dye) is poor in sulphonic acid groups, and therefore has little solubility in water. Copper compounds of substantive *o*-hydroxyazo dyes are not suitable, but certain chrome dyes may be used. Bluish-red, red, brownish-red, and black shades very fast to light and milling are obtained by dyeing wool with bis-(2 : 3-hydroxynaphthoyl)-1 : 5-naphthylenediamine-disulphonic acid, 2-benzamido-3-hydroxynaphthalene-6-sulphonic acid, the azo dye obtained by coupling diazotised 5-nitro-2-amino-*p*-xylene with 2 : 8 : 6-aminonaphtholsulphonic acid, or the dye obtained from diazotised *o*-aminophenol-*p*-sulphonic acid and 1 : 5-dihydroxynaphthalene, and coupling with diazotised *m*-xylidine. A. J. Hall.

[Machines for] dyeing silk or other material [in the form of skeins]. C. Callebaut and J. De Blicquy (E.P. 260,309, 28.4.25).—The machine comprises a horizontal elliptical cage formed by a number of rods eccentrically arranged around a rotating shaft. The axes of the cages are capable of adjustment so that tension on the skeins may be varied. During dyeing the skeins rotate so that not more than four points along the skein rotate in equal circular paths. A. J. Hall.

Dyeing and printing cellulose esters. I.-G. Farbenind. A.-G., Assees. of R. Metzger (U.S.P. 1,602,695, 12.10.26. Appl., 4.3.26. Conv., 6.3.25).—Anthraquinone-sulphamic acids give, on cellulose ester fabrics, fast shades, which can be improved by an after-treatment with a diazo-compound, or by diazotisation and treatment with an azo-dyestuff coupling component. For example, a cellulose acetate silk, immersed at 70—80° in a bath containing sodium anthraquinone-1 : 4-disulphamate, sodium sulphate, and sulphuric acid, is dyed reddish-violet shades. If formic acid is used in the bath, lighter shades are obtained. 4 : 8-Dihydroxyanthraquinone-1 : 5-disulphamic acid leads to reddish-blue, and 4-methoxyanthraquinone-1-sulphamic acid to bluish-red shades.
T. S. Wheeler.

Treatment of threads, fabrics, or other materials composed of or containing artificial [cellulose acetate] filaments. Brit. Celanese, Ltd., C. W. Palmer, and S. M. Fulton (E.P. 260,312, 26.5.25).—The lustre of cellulose acetate silk yarns is modified by treatment, in the presence of suitable quantities of protective salts (cf. E.P. 246,879; B., 1926, 317), with hot or boiling aqueous liquors which may or may not contain delustring agents, or with steam. Cane sugar is a protective substance additional to the neutral salts described in the earlier patent. A. J. Hall.

Weighting silk. J. Roscow (U.S.P. 1,602,840, 12.10.26. Appl., 15.7.25).—Silk is impregnated with a solution containing a barium salt, then dried and treated with a solution of a sulphate. A. J. Hall.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Acidic properties of silicic acid and certain silicates. H. Kappen and J. Breidenfeld (Z. Pflanz. Düng., 1926, A7, 174—190).—No decomposition of neutral salts, as shown by the development of exchange acidity, was found in the case of purified silicic acid. On treating silicic acid with barium hydroxide solution,

removing the excess of barium hydroxide by washing, and treating with aluminium chloride or ferrous chloride, silicates were obtained which possessed exchange acidities, corresponding to the amount of aluminium or iron, respectively, liberated by treatment with neutral salt solutions. Only a slight exchange acidity was produced by treatment with ferric chloride. These and further experiments are adduced in support of the view that in the case of natural and artificial zeolitic silicates, the acidity developed by reaction with neutral salt solutions is a consequence of direct exchange of aluminium ions against the cations of the salt solutions, and that there is no direct decomposition of neutral salts by the silicates in question. G. W. ROBINSON.

Titrations with arsenious acid. O. CANTONI (Annali Chim. Appl., 1926, **16**, 439—442).—The titration of arsenious acid by permanganate (B., 1926, 663) may be improved by substituting for potassium iodide a very dilute solution of iodine (prepared by oxidising a 0·002% solution of potassium iodide by permanganate). Manganese dioxide, lead dioxide, and triplumbic tetroxide may be determined by heating with arsenious acid and a strong acid, and titrating the residual arsenious acid with permanganate. E. W. WIGNALL.

The ammonia–soda process. E. TOPORESCU (Bull. Acad. Sci. Roumaine, 1926, **10**, 109—117).—The author has investigated the conditions of crystallisation of sodium bicarbonate in the ammonia–soda process by determining the solubilities at 15°, 35°, and 50° of sodium chloride, ammonium chloride, sodium bicarbonate, and ammonium bicarbonate, and also the conditions of equilibrium of mixtures of these salts in saturated solution at the same temperatures. The results are plotted on curves according to the method of Le Chatelier, so that the most favourable conditions for the manufacture of sodium bicarbonate can be deduced. The composition of the mixture giving the best yield can be regulated with accuracy, and the correct temperature for the decomposition of ammonium bicarbonate determined. W. G. CAREY.

Volumetric determination of magnesium in industrial waters. C. BELCOT (Bull. Soc. chim., 1926, [iv], **39**, 1483—1485).—Pfeifer's method for the determination of magnesium (Z. angew. Chem., 1902, **16**, 199), depending on the precipitation of magnesium salts by calcium hydroxide and determination of the excess of the latter reagent by titration with 0·1N-hydrochloric acid and phenolphthalein, has been tested for solutions of pure magnesium salts and for industrial waters containing iron. The method is rapid and sufficiently accurate for industrial work. A. S. CORBET.

Reduction of sodium sulphate to sodium sulphide. P. P. BUDNIKOV (Z. angew. Chem., 1926, **39**, 1398—1402).—Laboratory investigations showed that the conditions for optimum yield of sodium sulphide in the reduction of sodium sulphate by heating with carbon were as follows:—A temperature of 850°, a mixture of 2·0—2·5 pts. of pure sulphate to 1 pt. of carbon, and a time of melting of 40 min. With increase of time the yield rapidly falls off, and excess of carbon is injurious. Calcium oxide and ferric oxide as impurities are harmful; the oxides of magnesium and chromium have little effect. The process cannot be performed in an atmosphere of nitrogen, nor can the carbon be replaced by carbon monoxide or water-gas. C. IRWIN.

Separating nitrogen oxides from ammonia oxidation gas [by silica gel]. N. W. KRASE (Chem. Met. Eng., 1926, **33**, 674—679).—Liquid nitrogen tetroxide is easily handled in steel containers, and is a more convenient form of nitric nitrogen than nitric acid. The conditions for its extraction from ammonia oxidation gases were investigated in an experimental apparatus using pulverised silica gel in suspension in the gas stream. The gas was cooled and passed through an oxidation chamber into which excess of air was introduced. It then entered an adsorber tube fed with silica gel, and the treated gel was finally removed by baffles. The concentration of gel which could be kept in suspension was found to vary directly with the air velocity, inversely as the tube diameter, and to increase with the fineness of division of the gel. In all cases the maximum concentration possible is limited. Under practicable conditions one treatment with gel was found to remove 37—47% of the nitrogen oxides in the gas mixture, so that probably three successive treatments would be required. It is necessary, for this process, to remove water by previous cooling. Investigations with small aluminium towers fed with water showed that a time of contact of 1/70 sec. cooled the gas from 240° to 25° with a minimum loss of nitrogen as nitric acid. The gas then still contains 3% by volume of water vapour, and part of this is subsequently condensed as 60% nitric acid. The amount of this secondary loss depends on the time factor. C. IRWIN.

Oxidations and reductions of ammonium salts, nitrites, and nitrates by iron compounds insoluble in water. K. BORESCH (Z. Pflanz. Düng., 1926, **A7**, 205—232).—Certain materials containing iron in insoluble combination, including ferromagnesian minerals, iron ores, and basic slag, in a finely-ground state, can reduce neutral aqueous solutions of nitrates to nitrites. The reduction is increased by the presence of ammonium salts, which are themselves oxidised to nitrites. In these reactions ferrous oxide appears to act as a catalyst, and the formation of nitrites from nitrates and ammonium salts is an incomplete bimolecular reaction. Comparatively insignificant amounts of nitrite are produced by the action of manganese compounds. The significance of these reactions in the changes affecting nitrogenous compounds in the soil is emphasised. G. W. ROBINSON.

Determination of phosphoric acid. FRODL.—See XVI.

Determination of the toxic substance in insecticides. II. Volumetric determination of copper in insecticides containing iron, arsenic, and mercury. BODNAR and TERÉNYI.—See XXIII.

PATENTS.

Manufacture of oxalates and oxalic acid. W. WALLACE, Assr. to OLDBURY ELECTRO-CHEMICAL CO. (U.S.P. 1,602,802, 12.10.26. Appl., 23.3.20. Renewed 4.10.23).—A mixture of water and substantially equivalent quantities of calcium hydroxide and sodium oxalate are treated with carbon monoxide at 130° and 65 lb. pressure until absorption ceases. H. ROYAL-DAWSON.

Producing granular products [sodium hydroxide]. R. E. Wiley and C. E. Mensing (U.S.P. 1,601,897—8, 5.10.26. Appl., 9.7.25).—Molten sodium hydroxide is delivered into a rotating bowl, which throws it in the form of a spray, upwards and outwards, into a current of dry air carrying talc. The liquid solidifies in the form of dry, non-hygroscopic granules coated with talc, and falls in this form into a hopper surrounding the bowl. Means are provided for cooling the air employed, and using it again. T. S. Wheeler.

Refining or separating soluble substances [rock-salt] by crystallisation. Aktieselskapet Krystal, and F. Jeremiassen (E.P. 260,133, 5.1.26).—Approximately saturated solution (brine) is kept in circulation through the raw material and through a bed of granules of the crystallised material (sodium chloride). Before its passage through the raw material the solution is made just under-saturated by addition of steam, and therefore dissolves more salt. After passing through the raw material the liquid is made super-saturated, but only up to the meta-stable condition, by evaporation, and it then passes upward through the bed of pure granules which thereby increase in size. The raw material may be supplied through a closed hopper without stopping the circulation, and since the pure granules increase in size they will eventually become large enough to drop out of the rising stream of liquor, and may be removed at intervals. The evaporation is preferably produced by reduction of vapour pressure by means of a pump, and the steam withdrawn is pumped back into the liquid at its point of dilution. B. M. Venables.

Making aluminium halides and alkaline-earth-metal carbides. J. R. Mardick, Assr. to Union and Carbon Research Laboratories, Inc. (U.S.P. 1,600,899, 21.9.26. Appl., 21.7.23).—Aluminous material, an alkaline-earth halide, and carbon are heated together under such conditions that an aluminium halide volatilises from the charge, and an alkaline-earth carbide is obtained in a recoverable form. H. Royal-Dawson.

Chemical process [recovery of fluorides]. A. G. Betts (U.S.P. 1,598,672, 7.9.26. Appl., 9.1.22).—Crude calcium fluoride is treated with aqueous ferric chloride, and the solution obtained reduced electrolytically to separate pure calcium fluoride. Alternatively, calcium fluoride is mixed with a solution of aluminium and ferric sulphates, the iron present reduced to the ferrous state, and the solution treated with sodium hydrogen sulphate and hydrofluoric acid, when sodium fluoaluminate precipitates, and, on treatment with lime, yields pure calcium fluoride and sodium aluminate. The process can also be applied to the recovery of aluminium from felspar rocks, by treating them with calcium fluoride and sulphuric acid. The aluminium present goes into solution as aluminium fluosulphate, which is then treated as above. T. S. Wheeler.

Forming metal compounds and mixtures involving phosphorus. W. Koehler (U.S.P. 1,599,618, 14.9.26. Appl., 28.2.23).—A mixture of finely pulverised metal and phosphorus is subjected to a predetermined pressure. H. Royal-Dawson.

Recovering sulphur. J. Jannek, Assr. to I. G. Farbenind. A.-G. (U.S.P. 1,599,363, 7.9.26. Appl., 21.12.25. Conv., 22.12.24).—Superheated steam is passed through masses containing sulphur at extremely high speed, and the sulphur separated from the steam. H. Royal-Dawson.

Production of chlorine and sulphate from alkali chlorides. Zellstoff-fabrik Waldhof, and E. Schmidt (E.P. 249,474, 22.12.25. Conv., 21.3.25).—When sulphur trioxide, mixed with air, acts on alkali chlorides at 300—600°, no melting of such chlorides takes place, nor does the gas evolved contain any sulphur dioxide along with the chlorine. H. Royal-Dawson.

Oxidising catalyst. J. C. W. Frazer (U.S.P. 1,602,404, 12.10.26. Appl., 23.9.24).—A manganous compound is treated with cold nitric acid forming a finely-divided porous manganese dioxide. H. Royal-Dawson.

Apparatus for the purification of zinc solutions. T. D. Campbell (U.S.P. 1,601,938, 5.10.26. Appl., 15.2.26).—An apparatus consisting of a tank, inside which is a series of interconnected compartments, successively increasing in cross-sectional area, the smallest having an inlet, the largest an outlet. H. Royal-Dawson.

Producing oxidised carbon (E.P. 255,068).—See XI.

VIII.—GLASS; CERAMICS.

Influence of moisture on the mixing of batches for potash-lead oxide-silica glass. M. Parkin and W. E. S. Turner (J. Soc. Glass Tech., 1926, **10**, 213—220).—For the best conditions for mixing a batch for a full crystal glass, a moisture content of not more than 4·1% was desirable, and optmium results were found with values lying between 3·2 and 4·1%. A. Cousen.

Effect of moisture on the melting, working, and other properties of potash-lead oxide-silica glasses of the "English crystal" type. E. M. Firth, F. W. Hodkin, M. Parkin, and W. E. S. Turner (J. Soc. Glass Tech., 1926, **10**, 220—229).—In a series of batches for a full crystal glass containing from 0·5 to 10% of moisture, the batch having 4·1% melted most readily and "fined" most quickly. Least trouble from wave was found with glasses from batches with 4·1 and 5·1% of water, and the former batch gave the best working results. With 10% of moisture very poor working qualities resulted. A. Cousen.

Opal glasses. J. W. Ryde and D. E. Yates (J. Soc. Glass Tech., 1926, **10**, 274—291).—From an *X*-ray examination of opal glasses from batches containing fluorides the compounds separating out were found to be calcium fluoride, sodium fluoride, or a mixture of these. With glasses containing about 13% Na_2O the ratio of calcium to sodium fluoride separating increased from 0 to 4 as the CaO of the glass increased from 1 to 9%. With 5% CaO and 8% Na_2O only calcium fluoride separated, but with increase of the Na_2O to 12% equal quantities of the two fluorides were obtained. One semi-translucent opal contained only bubbles of diameter 3 to 20μ. A non-fluoride glass gave faint lines not

identified, but not calcium phosphate. Microscopical examination indicated the number of particles per c.c. as varying from 10^{12} to 10^{14} with average diameters from $1 \cdot 3\mu$ to less than $0 \cdot 3\mu$. The difference between opal and opalescent glasses is dependent upon the size and number of the particles, and the scattering coefficient is, in general, roughly proportional to the inverse square of the wave-length (one glass containing large gas bubbles proved an exception). When in sufficiently thin section opal glasses, even when very dense, show opalescent effects. Infra-red radiation is almost completely transmitted by opal glass. The effect of particle size can be investigated from suspensions of shellac in water. Scattering may be almost complete with particles $1 \cdot 5\mu$ in diameter, whilst with a diameter less than $0 \cdot 3\mu$ only half the incident light is scattered, although the particles are 125 times as numerous. Consideration of absorption shows that for glasses of equal scattering power there is less absorption in the case of larger particles (about $1 \cdot 0\mu$). A. COUSEN.

Durability of lead glasses and the Peddle generalisation. O. KNAPP (J. Soc. Glass Tech., 1926, **10**, 294—299).—A clearer relationship between durability and composition is obtained by employing the molecular oxide composition of the glass. Results given by Peddle for alkali-lead glasses (B., 1921, 771 A) when so expressed correspond fairly closely (with few exceptions) to the relationship log. $W = \frac{1}{8}R_2O + 0 \cdot 9$. Results obtained by the powder test may give an error of up to 10% due to variability of grain size, and as a check, in the case of glasses lying far from the curve, the iodoeosin test should be employed. A. COUSEN.

Durability of glasses containing zinc. W. L. BAILLIE (J. Soc. Glass. Tech., 1926, **10**, 299—304).—In a critical analysis of the results of Mauri (B., 1925, 881) in which the presence of zinc oxide in glasses was deduced as rendering these specially liable to attack by superheated water, the author states that it is doubtful whether the experimental data can be regarded as posssessing more than roughly qualitative value. A. COUSEN.

Relationship between chemical composition and the resistance of glasses to the action of chemical reagents. I. V. DIMBLEBY and W. E. S. TURNER (J. Soc. Glass Tech., 1926, **10**, 304—358).—The durability of a wide range of glasses was determined by submitting 10 g. of glass, powdered to between 30- and 40-mesh sieves, to the action of water, $20 \cdot 24\%$ hydrochloric acid, $2N$-sodium carbonate, and $2N$-caustic soda. The glass was held in a 90-mesh platinum or silver bag and kept for 1 hr. in 500 c.c. of the boiling liquid, the loss of weight and, in the case of water, the loss of alkali being determined. In a series of simple sodium silicate glasses, increase of the Na_2O content rapidly broke down the resistance to water and acid, the glass $Na_2O,2SiO_2$ dissolving almost completely. Sodium carbonate gave parallel results, but caustic soda caused an increase of weight in the glass. Series of glasses of the molecular type $6SiO_2,(2-x)Na_2O,xRO$ (or R_2O_3 or RO_2) were compared amongst themselves and with one another, in the latter case comparison being effected between glasses containing equal numbers of RO molecules per 100 molecules of SiO_2. Towards water, the order of decreasing effectiveness in giving durability was ZrO_2, Al_2O_3, TiO_2, ZnO, MgO, PbO, CaO, and BaO, and to acid the order was ZrO_2, Al_2O_3, ZnO, CaO, TiO_2, PbO, MgO, and BaO. With sodium carbonate the least effective oxide was BaO, followed by PbO, MgO, and CaO. To caustic soda ZrO_2 gave very resistant glasses, TiO_2 poor ones, CaO being good, whilst ZnO occupied a mean position. In a series of glasses with substitution, by weight, of the type $(80-x)\%$ SiO_2, $x\%B_2O_3$, $20\%Na_2O$, a slight increase of durability was shown as x increased from 0 to 8, but from this point to $x = 15$ a slow, and beyond, a rapid, decrease of durability resulted as the silica was replaced by boric oxide. With a series $(90-x)\%$ SiO_2, $x\%$ B_2O_3, 10% Na_2O, the durability continuously decreased as the boric oxide content increased. These boric oxide glasses were decomposed by boiling hydrochloric acid, the oxides B_2O_3 and Na_2O being dissolved, leaving the silica as a swollen, hydrated mass. From results obtained by the foregoing method upon commercial glasses, specifications were drawn up for durability in the case of bottle, chemical, and lampworking glasses. A. COUSEN.

Resistance of glass to chemical attack. B. P. DUDDING and W. SINGLETON (J. Soc. Glass Tech., 1926, **10**, 358—363).—In a graphic analysis of durability results of glasses examined by Cauwood, English, and Turner (B., 1917, 873) and Cauwood and Turner (B., 1918, 692 A) the curves obtained indicated that, in the case of soda-lime-silicate glass, good resistance to boiling water resulted even with relatively high percentages of sodium oxide, providing that the molecular ratio of basic oxides to sodium oxide was of the order 1 : 1. With smaller proportions of sodium oxide it was necessary to have a higher ratio of basic oxide to produce equally effective stability. Alumina beneficially affected the stability of the glass. By plotting the relative durabilities of chemical glasses to alkali against the molecular contents of boric oxide per 100 mols. of silica, smooth curves resulted, giving an optimum ratio of 6—8 B_2O_3 : 100 SiO_2. The same glasses only gave smooth curves for results by acid attack by plotting the silica content against attack, indicating a minimum of 67% SiO_2 as necessary to secure good resistance toward acids. A. COUSEN.

Action of heat on the surface properties of kaolin. R. DUBRISAY (Compt. rend., 1926, **183**, 782—784).—Four samples of kaolin in different stages of dehydration were made from washed kaolin by the following treatments: (1) no treatment, (2) heated for two hours at 250°, (3) heated for two hours at 550—600°, (4) heated for two hours at 950—1000°. It was found that the higher the temperature to which the sample was heated, the less became its iodine-absorbing power, its heat of wetting by water and by ammonia solution, and its absorptive capacity for water and lime-water. M. E. NOTTAGE.

Influence of impurities on the temperature of paramorphic transformation of cristobalite. R. WEIL (Compt. rend., 1926, **183**, 753—755).—The raising of the transformation temperatures of natural cristobalites by the action of sulphuric acid above 100° is attributed to the solvent action of the acid for impurities which tend to lower the transformation temperature. It

does not occur with artificial cristobalites having high transformation temperatures. Hydrochloric and nitric acids are without effect. The transformation temperatures of the cristobalite of quartz and of transparent commercial silica are 230—260° and 175—210°, respectively, owing to the effect of impurities, whilst the vapour produced by heating salts of the alkali metals in a flame may lower the transformation temperatures of cristobalites about 20° if these are originally high. The low transformation temperatures of 170—220° are due to the presence of volatile substances, the alkali metals especially, and do not appear in artificial cristobalite, which is obtained at high temperatures (A., 1926, 665). Non-volatile impurities (calcium, iron, magnesium) are responsible for the variation observed at 220—260°.

J. Grant.

Patents.

Apparatus for the equalisation of the density of mixtures of liquid and granular material [sand]. Soc. Anon. des Manuf. des Glaces et Prod. Chim. de Saint-Gobain, Chauny, et Cirey (E.P. 243,744, 26.11.25 Conv., 28.11.24).—The mixture of liquid and granular or powered material (*e.g.*, sand for glass grinding) is first concentrated in an inclined settling tank having parallel intermediate plates upon which the solid matter collects. The concentrated mixture is then fed into a tank through valves, which are actuated by a float situated in the mixture and opening or closing the valves according to the density of the mixture. W. G. Carey.

IX.—BUILDING MATERIALS.

Consistence of cement pastes, mortars, and concrete. N. Davey (Dept. Sci. Ind. Res. [Building], Tech. Paper 5, 1926, pp. 51).—The " flow table " test is satisfactory for determining the consistence of neat cement pastes and mortars, whilst this or the slump test can be used in the case of concretes. The consistence depends on the percentage amount of water used in mixing, on the particle size of the ingredients, on the chemical and physical nature of the particles of cement and aggregate, and on the temperature and degree of aeration of the mixture. The consistence which produces maximum density gives maximum strength and minimum permeability. In practice, however, especially in reinforced-concrete work, this is too stiff a mixture for satisfactory working. B. W. Clarke.

Manufacture of Portland cement from marl. R. E. Kirk (Bull. Univ. Minnesota Eng. Sta. No. 4, 1926, 29, 92 pp.).—Portland cement of satisfactory quality can be manufactured from marl and clay; the use of modern methods and equipments enables the wet process—which is more suitable for marl—to compete with the dry process. For successful commercial operation, a supply of good quality marl sufficient for 30 years should be available. Owing to the high water content necessary for the slurry when using marl, marl can never compete with limestone as a raw material where adequate supplies of the latter are available. B. W. Clarke.

Patents.

Production of cement mixed with gypsum. C. Pontoppidan and S. Buntzen (E.P. 260,447, 4.1.26).—Finely-ground raw gypsum is added to cement or cooled clinkers, and ground together in a mill kept sufficiently cool to prevent the rise of temperature and conversion of the gypsum into plaster of Paris.

H. Royal-Dawson.

Treatment of loose or fibrous organic materials and manufacture of light forms of concrete. Broadway Trust Co., Ltd., C. D. Burney, and H. O. Weller (E.P. 258,665, 6.5.25).—Fibrous organic material, *e.g.*, sawdust, is impregnated with a solution containing chromic acid or a dichromate with or without the addition of an acid or a metallic salt, *e.g.*, a solution containing, approximately, 2½% by weight each of potassium dichromate and ferric chloride, in order to prevent the swelling of the material with moisture, and is moulded under pressure with cement and a filler consisting of powdered limestone or slate dust, in order to obtain a light form of concrete. B. W. Clarke.

Manufacture of moulded articles from fibrous materials. Austro-American Magnesite Co., and K. Erdmann (E.P. 258,731, 19.9.25).—Fibrous material is heated with Sorel magnesite cement at 180—220° in an air bath, so that salt solution is always present until hardening is complete. This is preferably carried out in a chamber heated electrically or otherwise through which the material is conveyed on bands so arranged that no air can penetrate the chamber, which becomes saturated with moisture. B. W. Clarke.

Rendering roads dust-free by treatment with a dust-binding medium. Nederl.-Ind. Spiritus Maatsch., and T. J. A. Jacometti (E.P. 258,408, 20.8.25).—The sugar-free residual liquor from the manufacture of alcohol by fermentation of beet sugar or cane sugar molasses is concentrated and treated with an alkali to form hygroscopic salts, and worked up with road-making materials during the construction or repair of roads, with or without the addition of other hygroscopic substances, such as calcium chloride or magnesium chloride, thereby rendering the road free from dust. If desired, the liquid may be evaporated to dryness or a very small volume, and mixed with water immediately before use. B. W. Clarke.

Sand and like washing machines. Brun & Cie. (E.P. 250,554, 16.3.26. Conv., 9.4.25).—The sand (or gravel) is conveyed through a rotary drum, supplied with washing water, by a series of short plates normal to the drum wall and arranged along multiple-screw threads in rows parallel to the axis of the drum. Each plate is so inclined that the whole is engaged in lifting the sand out of the water. The ends of each plate are bent in opposite directions parallel to the axis, and are secured by brackets to the drum wall. H. Holmes.

Preserving wood. H. D. Heckert (U.S.P. 1,602,577, 12.10.26. Appl., 6.5.25).—Wood, submitted to compressed air at about 40 lb./sq. in., is treated with liquid preservative, without releasing the pressure, at about 150 lb./sq. in. until the predetermined proportion of preservative taken up has penetrated the wood, after which it is submitted to a reduced pressure of about 15 in. of mercury for about 20 min., and further treated with preservative at 110 lb./sq. in. pressure until the remainder has penetrated. H. Royal-Dawson.

Preservative treatment of wood. A. Arent, Assr. to A. Arent Laboratories, Inc. (U.S.P. 1,602,959, 12.10.26. Appl., 14.12.25).—Wood is rendered fireproof, and resistant to decay, by superficial treatment with a solution of antimony trichloride in a saturated, aqueous solution of sodium chloride. T. S. Wheeler.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Hardening of steel. H. Hanemann (Stahl u. Eisen, 1926, **46**, 1585—1587).—Microscopical examination of martensite shows that this substance is not homogeneous; annealing at 700° reveals differences in carbon content, the cementite separating in isolated needle-shaped areas. Austenite and martensite in the same section contain different amounts of carbon. These results are explained by the existence of a metastable martensite system in the iron-carbon constitutional diagram. An ϵ form of iron exists below 685°. The presence of carbon depresses the temperature of formation of this phase and gives rise to a duplex $\epsilon + \gamma$ field. At 350°, a peritectoid reaction occurs between ϵ-iron containing 0·1% C and γ-iron containing 1·4% C to produce a new constituent η with 0·9% C. The properties of hardened steel are discussed in the light of this theory. The η constituent has greater hardness and specific volume than the ϵ and γ fields. The breadth of lines found in the *X*-ray spectrograph is attributed, not to the fine structure of martensite, but to the close proximity of the lines due to the ϵ and η phases. L. M. Clark.

Use of high-frequency induction furnaces in the production of special steels. F. Körber, F. Wever, and H. Neuhauss (Stahl u. Eisen, 1926, **46**, 1641—1649).—Small charges (up to 50 kg.) of special steels may be satisfactorily melted and refined in the high-frequency induction furnace very much more rapidly than in the usual crucible furnace, owing to the rapid circulation of the molten metal under the refining slags causing a rapid removal of the impurities. Thus, a high-carbon chromium steel may be decarbonised to any desired extent down to 0·01% C without serious loss in chromium; at the same time the sulphur and phosphorus are also removed to such an extent that a very malleable, stainless iron is produced free from any tendency to hot-shortness. Remelting under a lime–fluorspar slag containing carbon admits of the rapid re-introduction of any quantity of carbon required, and the resulting steel, although containing much less than the usual amount of manganese, is not hot-short. Further advantages of this form of melting are the entire absence of slag inclusions in the castings, and the great ease of working combined with satisfactory mechanical properties. The energy consumption is about 1 kw.-hr./kg. for refining, and 1·5 kw.-hr./kg. for melting. A. R. Powell.

Experiments with electric annealing furnaces. T. Stassinet (Stahl u. Eisen, 1926, **46**, 1537—1547).—A Heraeus electric furnace for annealing a charge of 2·25 tons of hoop or strip steel in a neutral atmosphere at a temperature of about 800° is described. The total heat loss was analysed into its constituents, the greatest being through the refractory and insulating linings. By a mathematical treatment the optimum ratio of the thickness of these linings was determined, and a new furnace designed involving this and other improvements. The surface area of the annealing chamber was increased by corrugations, the parts not carrying heating elements being better insulated than those with them. Pure hydrogen or nitrogen was used, and an advantage was found in making the neutral gas eddy. Oil and petroleum tend to deposit carbon on the refractories, and were removed from the charge as far as possible, together with water vapour present. The resistor windings could be rapidly replaced. The new furnace yielded a better product, as determined by the Erichsen test, the heat losses being reduced from 299 to 130 kw.-hr. The energy consumption per ton—for a slightly lower annealing temperature which, however, yielded better tests—was 179—190 kw.-hr., corresponding to a reduction of 30—40%. The cooling period of the charge was reduced 65—75%, and lower still by artificial cooling with water, so that the output of the furnace was 60—70 tons per month against 5—5·5 tons for the old furnace. At a medium charge for current the electric annealing furnace can compete with modern flame furnaces. T. H. Burnham.

Temper-brittleness of steel. L. Guillet and M. Ballay (Rev. Mét., 1926, **23**, 507—520, 605—617).—The theories of temper-brittleness in steel are discussed, and a bibliography of the literature is given.

L. M. Clark.

Embrittlement of black-heart malleable iron. R. D. Allen (Trans. Amer. Soc. Steel Treat., 1926, **10**, 630—637).—To investigate the embrittlement of black-heart malleable iron which arose during grinding and hot riveting, test bars ½ in. × ½ in. × 10 in. were obtained from three sources. One set was hammered, a second set being taken in the original condition, and the two lots were then heated to a temperature of 225—425°. The cold-work specimens were embrittled and broke with a white steely fracture. The second set was tough, showing the characteristic black fracture. If hammered within the above temperature range the bars were embrittled in the same way. Quenching the embrittled iron from 650° restored it, and castings which received this treatment were not afterwards embrittled by hammering and heating to a low temperature. The tensile strength in the original condition was 24 tons with an elongation of 13%, and rose to 29 tons with no elongation in the embrittled condition, but no change was found in structure or ball hardness. T. H. Burnham.

Heat of formation of cementite. G. H. Brodie, W. H. Jennings, and A. Hayes (Trans. Amer. Soc. Steel Treat., 1926, **10**, 615—629).—The carbide used was obtained by melting together Armco iron and graphite in proportions to give the eutectoid composition, heat-treating the iron–carbon alloy to give 100% pearlite structure, and electrolysing in a 5% hydrochloric acid solution with a platinum cathode. It contained 6·66% C and 93·34% Fe. The heat of combustion was determined in an Emerson oxygen bomb calorimeter mounted in a constant-temperature box. The fractions of the iron which burned to ferric oxide and magnetite were determined and the total heat which the free elements of the sample would have evolved by combustion was

calculated. The difference gave the heat of formation per gram of carbide, from which the value per g.-mol. was found to be −13,580 g.-cal. at 30°, which agrees with the work of Ruff within the experimental error.

T. H. BURNHAM.

Transformations of austenite and martensite in hardened steels. G. TAMMANN and E. SCHEIL (Z. anorg. Chem., 1926, **157**, 1—21).—When hardened steel is cooled continuously from 20° to −150°, the conversion of austenite into martensite commences at about −20°, and continues to −150°. The change occurs, however, to a greater extent near the surface than it does within the mass, possibly because of a retarding effect of the high pressures prevailing in the interior. The temperature at which the rate of transformation into pearlite commences to be appreciable is about 100° for martensite, and about 250° for austenite. From density measurements the values $0{\cdot}1275\pm0{\cdot}00015$, and $0{\cdot}1310\pm0{\cdot}0002$ are extrapolated for the specific volumes of the austenite and martensite, respectively, in a steel containing 1·72% C. The latter figure agrees satisfactorily with that calculated from crystallographic data, provided that the distribution of carbon in the lattice is assumed to be irregular, and not otherwise. Pearlite produced from martensite at 200° has a greater sp. vol. than the pearlite in soft steel, but the excess gradually decreases with rise of temperature, and ultimately vanishes at about 500°. A similar effect is observed with austenite.

R. CUTHILL.

Influence of gases on the properties of steels. L. GUILLET and A. ROUX (Compt. rend., 1926, **183**. 717—719).—Samples of commercial steels and of nickel annealed in a vacuum have been found to liberate gas mixtures of constant composition containing, principally, hydrogen and carbon monoxide (40%), carbon dioxide, nitrogen, and traces of methane. The rate of evolution of the gas in all cases is a maximum at 750—850°, and the amounts produced are 0·49—3·3, 0·48—0·67, and 0·36—0·53 of the volume of the sample, for steels containing 0·04, 0·13, and 0·44% C, respectively. Nickel liberates 22 times its volume of gas. The sample containing 0·04% C was more soluble in sulphuric acid, after annealing in a vacuum, than in air, and in all cases the former conditions produced a less coarse crystalline structure.

J. GRANT.

Mechanism of oxidation of iron with steam, air, and carbon dioxide at high temperatures. P. P. FEDOTÉEFF [with T. N. PETRENKO] (Z. anorg. Chem., 1926, **157**, 165—172).—The product of the oxidation of iron by steam at 1000—1100° changes continuously in composition from ferrous oxide to ferrosoferric oxide, although at any given time only one solid phase is present. It thus seems that both isomorphous mixtures and solid solutions of the oxides must be formed. The first effect of the steam is probably to form a layer of ferrous oxide on the surface of the metal, and this is quickly oxidised to ferrosoferric oxide, which is reduced to the ferrous state again by the neighbouring metal, after which oxidation again occurs, and so on. If the iron is oxidised by air instead of by steam, ferrosoferric oxide is probably formed as before, but is oxidised to ferric oxide before solid solutions can be formed. If steam also is present, solid solutions are again formed. Oxidation with carbon dioxide occurs in the same way as it does with steam.

R. CUTHILL.

Microscopical examination of metallic minerals. M. J. ORCEL (Rev. Mét., 1926, **23**, 537—551, 618—626).—Microscopical examination of polished sections of minerals, together with their reflective power, dispersion in media of varying refractive index, colour, rotation of polarised light, hardness, and susceptibility to attack by various chemical reagents, are considered.

L. M. CLARK.

Fume losses at the Sulphide Corporation's works at Cockle Creek, N.S.W. G. L. CLARK (Proc. Austral. Inst. Min. Met., 1925, [60], 115—126).—The average loss of lead in the fume from the blast furnaces at the above works is a little less than 1% of the total lead in the charge when the furnace is working with a cool top, but often increases to as much as 9—10% when the furnace is run with a hot top, *e.g.*, in smelting for antimonial lead. These figures indicate that a deep charge column is an efficient fume collector. Practically no gold is lost in the fume, and only 0·014% of the silver in the charge. The average fume produced contains 50—60% Pb, 9—10% Zn, 9—18% As, and about 1% Sb. The loss of lead in fume from the Godfrey roasters is 0·5% using gas as fuel, and 1·3% with coal, but the latter is reduced to the same figure as with gas if the fire-bridge is raised 3 in. The silver content of the fume tends to settle out in the flues rather than in the bags. The average loss of silver from the cupels treating retort bullion from the Parkes process is 0·192%. The loss of lead in the softeners and refiners is 0·168% and of silver in the softeners 0·03%.

A. R. POWELL.

Plasticity of metals. H. SHOJI. **Plasticity of metals at high temperatures.** H. SHOJI and Y. MASHIYAMA (Sci. Rep. Tohoku Imp. Univ., 1926, **15**, 427—441, 442—447).—See B., 1926, 752.

Magnesium: melting, alloying, and casting. R. DE FLEURY (Rev. Mét., 1926, **23**, 649—657).

PATENTS.

Steel alloy [for hot working]. J. W. WEITZENKORN, Assr. to MOLYBDENUM CORP. (U.S.P. 1,601,787, 5.10.26. Appl., 30.3.26; cf. U.S.P. 1,401,926; B., 1922, 106 A).—A steel alloy, suitable for the manufacture of tools working at a high temperature, *e.g.*, rollers for steel mills, contains 0·85—2·5% C, 1·15—3·00 Mn, about 1—1·25% Mo, and as low a percentage of S and P as possible.

T. S. WHEELER.

Detempering steel. E. J. LEWIS (U.S.P. 1,602,274, 5.10.26. Appl., 5.5.22).—Hard steel may be softened by heating it to 412° and then quenching at 65° in a solution containing soda ash and soap solution.

M. E. NOTTAGE.

Pickling, scaling, and removing rust from articles of iron and steel. F. W. WÜRKER (E.P. 260,456, 27.1.26).—Small quantities of organic compounds of aliphatic and aromatic hydrocarbons containing at least one carbonyl group, such as ketones, or alcohols, or a mixture of these, are added to the dilute mineral acid; by this means the duration of pickling is considerably shortened and the pickle acts only on the oxide layer.

M. E. NOTTAGE.

Non-ferrous [tool] alloy. W. A. Wissler, Assr. to Haynes Stellite Co. (U.S.P. 1,602,995, 12.10.26. Appl., 10.1.25; cf. U.S.P. 873,745, 1,057,432, and 1,057,828; B., 1908, 128; 1913, 493).—A non-ferrous alloy suitable for the manufacture of high-speed tools for cast iron contains 20—30% W, 20—30% Cr, 0·75—2·4% C, 0·4—2% B, and the remainder Co, and unavoidable impurities. T. S. Wheeler.

Treating brass condenser tubes [against corrosion]. H. L. Weston, R. H. Conkle, and W. F. Clay (E.P. 260,491, 8.4.26).—Corrosion of the tubes by sea-water may be prevented by lining them with an alloy consisting of equal parts by weight of tin, lead, and zinc. The cleaned tube, supported above a heater, is slowly rotated until the temperature reaches 100°. The molten alloy is then poured slowly through, and the speed of rotation greatly increased so that the alloy is spread by centrifugal force evenly over the inner surface. The lining so formed is very coherent, unaffected by sea-water, and hard enough to resist the action of abrasives. M. E. Nottage.

Metallurgical process [for copper ores]. W. E. Greenawalt (U.S.P. 1,602,795, 12.10.26. Appl., 31.1.25).—Copper ores are concentrated to a relatively small amount of high-grade sulphide concentrate and a large amount of low-grade concentrate. The latter is roasted and then leached with a suitable solvent for copper; the high-grade concentrate is decomposed by heat in a reducing atmosphere to form hydrogen sulphide, which is then used to precipitate copper from the leach solution. M. E. Nottage.

Manufacturing metallic filamentary material. Control of crystal development in refractory metals. Preparation of tungsten powders. W. B. Gero, Assr. to Westinghouse Lamp Co. (U.S.P. 1,602,525—7, 12.10.26. Appl., [A], 13.1.21, [B] 15.9.22, [C] 18.3.24. Renewed 2.4.26).—(A) Tungsten oxide, free from compounds which deleteriously affect grain growth, is mixed with at least one salt of an alkali or alkaline-earth metal capable of promoting a definite grain or crystal structure in a filament of the mixture when annealed or burned, and the mixture reduced in hydrogen. Filaments so prepared are resistant to sagging and offsetting when burned at high temperatures. (B) Tungsten obtained as under (A) is sintered and mechanically worked to filament size. The filament, composed of large crystals fairly regular in shape and size, may be coiled, and is then non-sagging and non-twisting. (C) Tungsten oxide is mixed with a compound of an alkali metal, and reduced by hydrogen. J. S. G. Thomas.

Precipitating hafnium and zirconium on an incandescent body. W. J. Tennant. From N.V. Philips' Gloeilampenfabr. (E.P. 260,062, 7.8.25).—Hafnium or zirconium is deposited upon a core by heating the core in an atmosphere containing one or more iodides of the respective metals at such temperature, *e.g.*, above 1750° in the case of the deposition of hafnium and above 1600° in the case of zirconium, that the metal precipitated grows in a direction at right angles to the surface of the core so as to form a single crystal. The iodides employed must be sufficiently pure to ensure that the precipitated metals are ductile.
J. S. G. Thomas.

Recovery and refining of precious metals. A. Gordon (E.P. 260,451, 11.1.26).—The platiniferous material (platinum concentrate) is fused with zinc or lead, the alloy so formed is treated with hydrochloric acid, and the insoluble constituents, viz., platinum metals, gold, and the chlorides of lead and silver, are separated from the solution. In the case of the zinc alloys, the zinc is recovered from the solution by adding metallic zinc which precipitates all the base metals except iron, which may be precipitated by adding zinc oxide or freshly precipitated zinc hydroxide and passing in chlorine; the solution, after removal of the precipitate, contains practically pure zinc chloride. From lead alloys a pure lead chloride solution may be obtained by extracting the insoluble residue with hot water. The zinc or lead chloride solution is then electrolysed with the production of pure zinc or lead, which may be used again, and chlorine, which may be used for the extraction of the precious metals. To separate the precious metals the metallic residue is suspended in water, and chlorine from the electrolytic plant is passed in; a solution of gold chloride is obtained from which gold may be extracted by known processes. The solid residue is treated with hydrochloric acid and more chlorine passed in; the palladium and platinum dissolve. Ruthenium may be recovered from the residue by fusing it with potassium hydroxide and nitrate, saturating the aqueous extract with chlorine, and distilling the ruthenium over in a stream of chlorine. M. E. Nottage.

Electric furnaces for bright annealing (E.P. 244,427). **Protecting cuprous metals** (E.P. 260,536).—See XI.

XI.—ELECTROTECHNICS.

Anodic oxidation of aluminium and its alloys as a protection against corrosion. G. D. Bengough and J. M. Stuart (Dept. Sci. Ind. Res., 1926, 40 pp.).—Anodic oxidation of aluminium, duralumin, and other aluminium alloys, in a bath consisting of a 3% solution of chromic acid (cf. E.P. 223,994—5; B, 1925, 14), results in the production of a thick, glassy, adherent film of oxide over the entire surface of the metal, even in deeply-recessed parts. This film effectually protects the metal from corrosion in salt water for prolonged periods; it is not destroyed by heating for 5 min. at 350°, so that it may be applied to the metal before annealing. The unheated film acts as a mordant to many basic dyes, and therefore admits of the production of coloured films on aluminium articles. Both coloured and uncoloured films may be waterproofed by impregnation with lanoline; after this treatment, duralumin remains unaffected for many months even in contact with stainless steel. The presence of alkaline substances in the corroding medium causes rapid disintegration of the ordinary film, but does not affect it after the grease treatment. The bath used for the electrolysis should be as free as possible from sulphuric acid, the presence of which tends to produce less adherent deposits. Before treatment the metal is cleaned with petrol, dried, and washed in hot water. The graphite cathodes used should have an area about double that of the aluminium anode. The temperature of the bath is raised to 40° and the voltage raised slowly from 0 to 40 volts during 15 min., the solution being kept

agitated. After a further 35 min. the voltage is increased to 50 during 5 min. and maintained at 50 for another 5 min. The article is then washed and dried. The current consumption varies according to the nature of the material from 3—6 amp./sq. ft., and the average cost is about 2d./sq. ft. A. R. Powell.

Applications of X-rays in metallurgy. J. J. Trillat (Rev. Mét., 1926, 23, 671—684).

Electrodialysis of the colloidal soil material and the exchangeable bases. Mattson.—See XVI.

Patents.

Electric furnaces for bright annealing. Siemens-Schuckertwerke Ges.m.b.H., Assees. of Heraeus-Vacuumschmelze A.-G., and W. Rohn (E.P. 244,427, 28.10.25. Conv., 15.12.24).—In an electric bright-annealing furnace for continuous working (cf. E.P. 242,283; B., 1926, 834) an inner heat recuperation process is obtained by causing the two lines of articles to be heated to travel through the furnace in opposite directions, and in separate tracks, each of which is a heating chamber with adjoining ante-chambers. M. Cook.

Electrolytic preparation of silver halides from metallic silver. S. E. Sheppard and R. H. Lambert, Assrs. to Eastman Kodak Co. (U.S.P. 1,602,595, 12.10.26. Appl., 27.2.26).—Anode silver is electrolytically converted into silver halide in an aqueous electrolyte which is a solvent of the silver halide and contains halide anions. The solvent power of the electrolyte is reduced, and the silver halide dissolved during the first step is precipitated. For the precipitation of silver bromide, for example, a suitable electrolyte is a hot, preferably saturated, solution of potassium bromide, maintained slightly acid by hydrobromic acid. W. Clark.

Method of producing oxidised carbon [graphitic oxide]. C. F. Burgess Laboratories, Inc., Assees. of B. K. Brown, O. W. Storey, C. A. Silver, and G. T. Collinson (E.P. 255,068, 7.7.26. Conv., 7.7.25).—Graphitic oxide, of dull black appearance, but chemically similar to the yellow product, is prepared by the electrolysis of 30% nitric acid to which 5% of sodium dichromate has been added, using a carbon anode. The oxide is formed as a sludge at the bottom of the cell, and contains from 1% up to 5—6% of available oxygen as determined by ferrous sulphate. The current density should be below 10 amp./sq. ft. and the temperature 22—25°. The anode is prepared from graphite crushed to pass a 10-mesh screen, moulded with rosin as a binder, and impregnated with paraffin or the like. The powdered product is washed with water and benzene and dried at a temperature below 100°. It is prepared for use as a depolariser in a dry cell. C. Irwin.

Determining and indicating the electrical conductivity of fluids. R. E. Gillmor. From S. Ruben (E.P. 260,366, 8.8.25).—In apparatus for the determination of the electrical conductivity of fluids, more especially boiler feed water, one of the electrodes employed is mounted on a bi-metallic strip, so that the distance between the electrodes varies with the temperature of the electrolyte, and thus compensates for temperature effects. The graduations of the iron-core ammeter used as indicator are closer together for high current values than for low. J. S. G. Thomas.

Protection of cuprous metals against corrosion. G. B. Ellis. From T. E. Murray (E.P. 260,536, 24.6.25).—Metals such as copper and brass are electrolytically coated first with a layer of ferrous metal, and then with one of chromium. Aluminium may also be used as a third coating metal. The different coatings are diffused into each other in the solid state, either by a preliminary heating at a high temperature or by the temperature attained in actual use. M. Cook.

Electrolyte for electrolytic [rectifying] cells. J. Slepian and E. J. Haverstick, Assrs. to Westinghouse Electric and Manuf. Co. (U.S.P. 1,602,951, 12.10.26. Appl., 9.12.19).—The addition of about 1% of an electrolyte, with no rectifying action, and of an alkali to the electrolytes ordinarily used in rectifying cells increases the life and improves the power factor of the cell. A preferred electrolyte comprises, per litre, boric acid 30 g., ammonium borate 5 g., sodium hydroxide 2—10 g., and sodium fluoride 0·5—3 g. T. S. Wheeler.

Electrolytic apparatus. H. P. Ewell (U.S.P. 1,599,701, 14.9.26. Appl., 19.10.25).—A solution of a metallic salt is circulated in counter-current to a stream of mercury in a tank provided with means for decomposing the metallic salt electrolytically. J. S. G. Thomas.

[Electrodes for] batteries, electric accumulators, and electrolysing apparatus. Soc. Anon. le Carbone (E.P. 244,417, 22.9.25. Conv., 10.12.24).—Absorbing and catalysing material, *e.g.*, silica gel or dehydrated alumina gel, rendered completely impermeable to liquids by exposure to hydrocarbon vapours, or by mixing with rubber solution, is incorporated in the material of the electrode, *e.g.*, lead. J. S. G. Thomas.

Electron discharge apparatus [high-frequency signals detector]. The British Thomson-Houston Co., Ltd., Assees. of E. E. Charlton (E.P. 237,276, 17.7.25. Conv., 17.7.24).—A high-frequency detector comprising a triode valve containing an alkali metal, preferably cæsium, and having a cathode of a material, *e.g.*, thoriated tungsten, which when heated above a critical temperature of about 1000—1200°, under operating conditions, emits both electrons and positive ions, so that when the device is operated at room temperature, with the cathode heated above the critical temperature, the positive ions produced enable the device to function sensitively as a detector. J. S. G. Thomas.

Incandescent lamp. P. A. Campbell, Assr. to General Electric Co. (U.S.P. 1,600,203, 14.9.26. Appl., 2.3.22).—Solid carbonaceous material is applied to the surface of an incandescent non-carbonaceous refractory material contained in a bulb, in such quantity as to prevent discoloration of the bulb during the early part of the life of the lamp without substantially changing the physical properties of the refractory material. J. S. G. Thomas.

Moulded phenolic [insulating] compositions. G. L. Peakes, Assr. to Bakelite Corp. (U.S.P. 1,602,249, 5.10.26. Appl., 19.11.23).—Insulating materials, formed from a cured, infusible, moulded phenol resin and a

filler, such as wood, have their insulating powers, especially at elevated temperatures (100°), greatly improved by heating at 125—135° for 48—80 hrs.

T. S. WHEELER.

Recovery and refining of precious metals (E.P. 260,451).—See X.

XII.—FATS; OILS; WAXES.

Maize oil. F. WILBORN (Farben-Ztg., 1926, **32**, 240—242).—A sample of maize oil pressed from a mixture of equal proportions of La Plata and S. Russian seed (both 1924 crop) had d^{15} 0·921, saponif. value 190, and iodine value (Hanus) 121. The oil had characteristic smell, reminiscent of goose fat, darkened at 200°, and "broke" at 250°, the mucilage content being rather high. The fresh oil dried very slowly, not setting in 1 month, but after keeping for 7 months a drying time of 21 days is recorded. Maize oil paints dried in from 2 to 42 days, depending on oil absorption and nature of the pigment used. The drying curves of the oil with and without the addition of driers (cobalt linoleate), and when heated for 10 hrs. at 300° in an atmosphere of carbon dioxide to give the corresponding stand oil, are shown, all exhibiting an initial rise and fall before the usual increase to maximum weight. Drying times of linseed oil–maize oil–driers mixtures are tabulated, *e.g.*, a 60 : 40 mixture + 5% of lead–manganese rosinate drying in 7½ hrs. Maize oil itself + 5% of driers sets in about 15 hrs. The stand oil previously mentioned has d^{15} 0·939, iodine value 83·2, and gives a soft, tacky film, but satisfactorily drying enamels may be made with it, which do not show the yellowing on ageing that is a drawback of the stand oils from linseed oil and tung oil.

S. S. WOOLF.

Highly unsaturated acids of Japanese sardine oil. M. TSUJIMOTO (Chem. Umschau, 1926, **33**, 285—291).—The fatty acids, m.p. 32°, saponif. value 195·0, iodine value (Wijs) 174·9, polybromides 58·2%, from a Japanese sardine oil (d^{15}_4 0·9390, acid value 3·3, saponif. value 189·2, iodine value 177·5, n^{20} 1·4800, unsaponifiable matter 1·01%) yield, by the lithium salt–acetone method (B., 1920, 825 A), 33·8% of highly unsaturated acids (n^{20} 1·4913, saponif. value 171·0, iodine value 356·5). By fractional distillation of the methyl esters of the highly unsaturated acids, it is shown that the chief constituent is an acid having the formula $C_{22}H_{34}O_2$, which is proved to be the correct formula for clupanodonic acid (cf. Schmidt-Nielsen, B., 1922, 300 A; Brown and Beal, B., 1923, 665 A). The acid $C_{18}H_{28}O_2$ is present and probably the acids $C_{20}H_{30}O_2$ and $C_{20}H_{32}O_2$. There samples of clupanodonic acid have been prepared from the highly unsaturated acids isolated from the oil by the bromide method, the lithium salt–acetone method and the modified lithium salt–acetone method (B., 1924, 62), and the following constants are the mean values for the acid and its methyl ester respectively: d^{15}_4 0·9385, 0·9246; d^{20}_4 0·9356, 0·9204; n^{15} 1·5039, 1·4958; n^{20} 1·5020, 1·4943. The lithium salt–acetone method is the most suitable for the separation of the highly unsaturated acids, especially for clupanodonic acid.

E. H. SHARPLES.

Equilibria underlying the soap-boiling processes. System potassium laurate–potassium chloride–water. J. W. McBAIN and M. C. FIELD (J. Physical Chem., 1926, **30**, 1545—1563).—Phase diagrams for the systems potassium laurate–water, and potassium laurate–potassium chloride–water at 18°, 90°, and 200° have been worked out. These systems are similar to those of other soaps, such as potassium oleate and sodium palmitate (cf. McBain and Elford, A., 1926, 358), the chief phases being crystals, isotropic liquid, and two anisotropic liquids, viz., middle soap and neat soap. All the phases found in the three-component system which includes salt, occur in the two-component system, soap and water. The limits of the field of existence of isotropic liquid solutions of potassium laurate have been determined up to 376° in the absence of the chloride and up to 225° in its presence. This single phase includes solutions which are wholly crystalline as well as those which are colloidal electrolytes, and ranges, at sufficiently high temperatures, from pure water up to anhydrous potassium laurate, complete miscibility occurring above 376°. The neat soap and middle soap are typical conic anisotropic liquids, and the latter is much more prominent with potassium laurate than for the higher soaps, and more likely to disturb the normal course of soap making.

L. S. THEOBALD.

Determination of free alkali in soaps. J. DAVIDSOHN (Chem. Umschau, 1926, **33**, 273—281).—The author reviews the various methods of determining free alkali in soap, and the results of numerous determinations on pure soaps to which known amounts of soda or potash were added are tabulated. The conventional alcohol-extraction method is shown to be untrustworthy when filtration is introduced, though results closely in agreement with the theoretical are obtained when this operation is avoided. The methods of Heermann, Bossard and Huggenberg, and Ismailsky (B., 1926, 923), with their various modifications, have been experimentally investigated by the author, who finds them unreliable. Reviewing methods based on salting out the soap by sodium chloride and subsequent titration of free alkali in the filtrate, the author recommends the following, based on the use of anhydrous sodium sulphate: (A) For hard soap, 3—5 g. of soap are dissolved (refluxed) in 50—70 c.c. of warm 95% neutralised alcohol and after cooling titrated with $N/10$ hydrochloric acid, using phenolphthalein as indicator, and calculated to sodium hydroxide. (B) For soft soap, 3—5 g. are dissolved as for hard soap, and to the solution is added slowly in small portions 4—6 g. of pure anhydrous sodium sulphate. Then, with phenolphthalein as indicator, titrate with $N/10$ alcoholic hydrochloric acid and calculate to potassium hydroxide. For a strongly hydrated soap, method (B) is used.

H. M. LANGTON.

Animal skin fat. McLAUGHLIN AND THEIS.—See XV.

Leather japans. ENNA.—See XV.

Detergent action of soap. WOODMAN.—See XVI.

PATENTS.

Method of obtaining scalp-treating oils. A. P. ELIADES (U.S.P. 1,602,004, 5.10.26. Appl., 26.6.24).—The whole meats of butternuts are soaked in brine, roasted, and after comminuting mixed with a quantity of pre-

viously extracted butternut oil and water. The mixture is heated to boiling and, after draining the free oil from the pulp, the residue is pressed. E. H. Sharples.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Fluorescence-analysis of oil varnishes. H. Wolff and W. Toeldte (Farben-Ztg., 1926, **32**, 80—81).—Several experimental varnishes were examined for fluorescence phenomena, under the dark-filter mercury vapour lamp, at progressive stages in their drying, the latter being carried out (*a*) under normal conditions, (*b*) by stoving at 100—110° after 17 hrs. air-drying, and (*c*) by stoving at 100—110°. The conditions of drying and the presence or absence of driers were found markedly to influence the phenomena and the differences between varnishes containing Congo copal, rosin (colophony), ester gum, and Albertol as resin components were much slighter than the observed differences in the characteristic fluorescences of the resins themselves (cf. B., 1926, 796). S. S. Woolf.

Evaluation of [water] paint adhesives. H. Wagner (Farben-Ztg., 1926, **32**, 182—186, 242—245).—An account of the physical and chemical testing of glues, starches, casein preparations, sodium silicate, etc. in relation to their use as water-paint binding materials. The suitability of the various types under particular painting conditions is indicated. (Cf. Wagner, B., 1926, 638.) S. S. Woolf.

Storch–Morawski [Liebermann] reaction for abietic acid. M. Schulz and F. Krämer (Farben-Ztg., 1926, **32**, 82—83, 303—304).—Polemical with E. Stock (see following abstract), on the validity of the Storch–Morawski reaction, particularly in connection with Albertol resin varnishes. (Cf. also B., 1926, 796.) S. S. Woolf.

Storch–Morawski [Liebermann] reaction for abietic acid. E. Stock (Farben-Ztg., 1926, **32**, 303).—Polemical. (See preceding abstract.) S. S. Woolf.

Maize oil [for paints]. Wilborn.—See XII.

Leather japans. Enna.—See XV.

Volatile oils from *Xanthorrhœa* species. Finlayson.—See XX.

Patent.

Moulded phenolic [insulating] composition (U.S.P. 1,602,249).—See XI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Significance of the resin of *Hevea* rubber in vulcanisation and in the ageing of raw rubber. G. S. Whitby and H. Greenberg (Ind. Eng. Chem., 1926, **18**, 1168—1171).—The "resin" acids in *Hevea* rubber have an important influence on the rate of vulcanisation in the presence of accelerators; the addition of stearic acid also improves the vulcanisation of Caucho and *Ficus* rubbers, which are relatively deficient in natural "resin" acids. The natural acid content of *Hevea* rubber decreases on ageing, probably by the oxidation of unsaturated members of the "resin" acids. The non-acid constituents of the "resin" have little influence on vulcanisation. D. F. Twiss.

Optimum cure [vulcanisation] criteria in rubber. W. B. Wiegand (Ind. Eng. Chem., 1926, **18**, 1157—1163).—The various methods for the determination of the optimum degree of vulcanisation are reviewed. In view of the necessity to avoid undesirable proneness to rapid ageing, the degree of vulcanisation for technical products is generally less than that yielding the immediate optimum characteristics; the difference commonly is greatest for materials containing rubber with plenty of sulphur and little accelerator, and least for highly accelerated "compounds" containing a small proportion of sulphur. D. F. Twiss.

Present and future of reclaimed rubber. H. A. Winkelmann (Ind. Eng. Chem., 1926, **18**, 1163—1168).—In the "reclaiming" of scrap vulcanised rubber the disaggregating effect of heat is probably the most important factor, although aided by various softening agents. The caustic alkali commonly used in reclaiming definitely aids the production of a plastic product. The amount of chloroform-soluble material yielded after extraction with acetone probably provides an indication of the relative value of reclaimed rubber. The proportion of sulphur in the material removed by chloroform is much lower than that in the undissolved portion. D. F. Twiss.

Patents.

Producing rubber and like solutions. A. Fraser, Rissik, Fraser & Co., Ltd., and F. Shaw & Co., Ltd. (E.P. 259,649, 1.7.25).—An apparatus for producing continuously rubber, balata, gutta, or similar solutions comprises a steam-heated cylinder containing a rotating feed screw, by means of which rubber is forced in a thin tubular film into a centrifugal grinding machine, formed by a stationary and a rotating disc. Means are provided for introducing solvent and vulcanising liquids to the space between the discs. T. S. Wheeler.

Manufacturing [sponge] rubber goods. C. O. North, Assr. to Rubber Service Laboratories Co. (U.S.P. 1,602,624, 12.10.26. Appl., 19.3.23).—In the manufacture of sponge rubber plasticised rubber is treated, in the mixing mill, with a suspension of 15 pts. of ammonium carbonate in 8 pts. of a mineral oil of b.p. above 100°. No evolution of ammonia occurs until the mixture is heated in the moulds to vulcanise the rubber. Mineral oil can also be employed to incorporate accelerators or the like. T. S. Wheeler.

Mastication of rubber and apparatus therefor. Dunlop Rubber Co., Ltd., H. C. Young, and H. O. Burr (E.P. 260,310, 1.5.25).—Rubber passes between the rolls of an ordinary masticating mill on to an endless conveyor, which returns the rubber to the mill. While on the conveyor the rubber is subjected to the cooling action of a current of cold air. D. F. Twiss.

Devulcanising vulcanised rubber and the product thereof. C. F. Willard (U.S.P. 1,602,062, 5.10.26. Appl., 19.11.23).—Vulcanised rubber is boiled with an emulsoid colloid solution and a sulphur solvent which is also capable of dissolving rubber. The operation is continued until the devulcanised rubber passes into solution. D. F. Twiss.

Preparation of an anthelmintic [from latex] (E.P. 243,325).—See XX.

XV.—LEATHER; GLUE.

Animal skin fat. G. D. McLaughlin and E. R. Theis (J. Amer. Leather Chem. Assoc., 1926, **21**, 551—559).—Skin fat consists of true fats, glycolipins, phospholipins, unsaponifiable matter mostly cholesterol, and hydroxy-fatty acids. Extraction of skin with fat solvents removes a certain definite amount of fat which is increased by the addition of acetic acid to the solvent. Apparently some of the lipin is in the free state, and some is combined with the skin proteins and requires hydrolysis for its extraction. This hydrolysis can be brought about by bacteria. The amount of fat extracted from different kinds of skins shows that the fat is present in both the corium and epidermis in amounts varying with the age and sex of the animal. The amounts in the corium of mature steer, young steer, heifer, and bull hides were 65%, 11%, 82%, and 14% respectively of the total extractable fat in the whole of the skins. Post-mortem changes affected the amount and character of the extractable fat, increasing it by 13% for fresh corium which had been left 48 hrs. After 72 hrs. the extractable fat was twice as rancid. Prompt curing of the skins prevented or minimised such post-mortem changes. Soak liquors favourable to proteolytic bacterial digestion increased the amount of fat, probably owing to the decomposition of skin proteins which contain fat. A lime liquor saponified 25% of the extractable fat in a hide, and 40% from cubes of hide which presented three times the surface of the uncut hide. An excess of sodium or calcium hydroxide was found necessary to produce saponification of the extractable fat. *N*/20-Sodium hydroxide did not saponify the fat in the hide more quickly than did *N*/2-calcium hydroxide, although it had more rapid action on the extracted fat. The difference is attributed to the greater swelling effect of the caustic soda causing the skin to be more compressed, thus hindering saponification of the fat in the skin.

D. Woodroffe.

Employment of some iron compounds as driers of leather japans. Studies in patent leather. II. F. G. A. Enna (J. Soc. Leather Trades Chem., 1926, **10**, 311—326; cf. B., 1925, 370).—Samples of linseed oil were heated under various conditions. Without the addition of a drier, the colour of the boiled oil was light yellow. The addition of water to the oil during heating accelerated polymerisation, but did not affect the colour or drying power. The addition of 10·9% of ferric chloride caused the oil to become green at 60°, and to evolve hydrochloric acid gas at 100—110°. The polymerisation proceeded at a much faster rate and at a lower temperature, and the japan produced was very dark brown. The addition of hydrochloric acid, ferrous oxalate, and of other substances, yielded products which showed that the iron is chiefly responsible for the drying properties exhibited by oils when heated with its compounds, the valency of the iron being immaterial. The speed of polymerisation of the oil heated with iron salts depends on the solubility of the latter in the oil and on the total quantity present. The colour of the japan, apart from that imparted by the iron, is influenced by the acid with which the iron is combined. The addition of water brings about an initial, though slight, dissociation of the glycerides. Mineral acids tend to darken the colour of the boiled oil and retard the speed of drying, when the acid is a strongly dissociated one. D. Woodroffe.

Nitrogen content of hide substance. M. W. Kelly (J. Amer. Leather Chem. Assoc., 1926, **21**, 573—583).—The moisture in hide powder, as determined by heating in an air oven at 100°, decreased with increasing time of heating, whilst by heating for 16 hrs. in a vacuum oven at 100°, the results were equally satisfactory, but about 4% higher. The ash of hide powder was determined by calcining a sample in a platinum dish in the electric muffle. When the fat was extracted from hide powder with different solvents, light petroleum gave the least extract, carbon disulphide more, and chloroform the most. The increased amount of extract with chloroform was not due to protein. The nitrogen was determined by the official method of the American Leather Chemists' Association for Kjeldahl determination, except that 0·3 g. of copper was added as catalyst during the digestion. The results were 15·45—15·99% for hide powder and 14·83—15·56% for corium. The nitrogen, on the moisture-, fat-, and ash-free basis, varied from 17·88 to 18·06% for American standard hide powder, and from 17·76 to 17·91% for corium from fresh steer hide, as compared with 17·80% as determined by von Schroeder and Paessler.

D. Woodroffe.

Determination of sulphur dioxide in bleaching [tannin] extracts. D. Burton and H. Charlton (J. Soc. Leather Trades Chem., 1926, **10**, 326—333).—3·5—4·0 g. of the extract are weighed into a special distilling flask connected with a series of three wash bottles containing, in the first two, standard iodine solution, and potassium iodide solution in the last. The apparatus is devoid of any rubber connexions; 200 c.c. of water are added, and the free sulphur dioxide is determined by distillation in a current of carbon dioxide for 30 min. and about 30 c.c. of water are distilled over. The residual iodine is determined in the three bottles, and the free sulphur dioxide calculated therefrom. 10 c.c. of concentrated phosphoric acid (*d* 1·750) and about 50 c.c. of water are added to the distillation flask and distilled for a further 30 min. in a current of carbon dioxide to liberate the combined sulphur dioxide, which should be collected in a measured quantity of standard iodine solution as before. The amount of combined sulphur dioxide can be calculated from the iodine used. Experiments with sodium sulphide have demonstrated the superiority of phosphoric acid over other acids. The free sulphur dioxide is an indication of the immediate and the combined sulphur dioxide of the lasting bleaching properties of an extract. The ratio is an index of the value of the bleaching extract.

D. Woodroffe.

Variable tannin content of wood of Kumaon oak (*Quercus incana*). H. S. Chaturvedi and E. R. Watson (J. Indian Chem. Soc., 1926, **3**, 211—212).—The percentage of tannin in the wood of this tree is small and variable, and it has no economic importance.

G. M. Bennett.

Analysis of synthetic tannins. R. Escourrou (Chim. et Ind., 1926, **16**, 373—375).—Solutions of "cleartan" when evaporated for total water-soluble matter, charred even at 90°, giving an insoluble residue.

Solutions of quebracho extract, "maxyntan," and "cleartan" were evaporated on the water bath and heated in an oven at 90°. Curves are given which show that the quebracho dried to constant weight in 7 hrs., whereas the others were still losing weight after 32 hrs. The loss in weight is probably due to further condensation or polymerisation processes. Better results were obtained by drying *in vacuo*, and this method should be applied in evaporating and drying the filtered solutions of syntans in determining water-soluble matter. It requires 7—8 days, but it can be speeded up by applying very moderate heat. The non-tans do not require to be treated in this way, as the substances liable to polymerise are usually absorbed by the hide powder.

D. WOODROFFE.

Examination of artificial horn made from casein. J. OBRIST and O. MANFRED (Z. Angew. Chem., 1926, **39**, 1293—1300).—Exhaustive examination in polarised light of sections of artificial horn, manufactured by the ordinary methods, leads to the conclusion that the double refraction observed affords no indication as to either the homogeneity of the material or the method of manufacture. A large number of photo-micrographs are reproduced. S. I. LEVY.

Evaluation of [water-]paint adhesives. WEIGNER. —See XIII.

PATENTS.

Varnishing [patent] leather. J. PAISSEAU (E.P. 255,803, 27.10.25. Conv., 23.7.25).—The surface of the leather may be gelatinised either by applying a dilute solution of a volatile organic or other acid, or a thin coat of gelatin which is subsequently rendered insoluble by tanning or other process. A varnish of cellulose nitrate or acetate is then applied, containing 5—10% of acetic acid if the surface has not been gelatinised, and a substance conferring plasticity, such as castor oil or a stable, non-volatile, non-resinous derivative. Two or three coats of the varnish are applied, pearl essence being added to produce silky, velvety, lustrous or pearly effects. D. WOODROFFE.

Tanning material. J. K. TULLIS (U.S.P. 1,603,169, 12.10.26. Appl., 17.3.22).—Hides are treated with a solution of 2 pts. of chromic acid, 5 pts. of magnesium sulphate, and 4 pts. of aluminium sulphate.

D. WOODROFFE.

Casein glue. H. L. PRESTHOLDT (U.S.P. 1,604,307—17, 26.10.26. Appl., 17.4.22).—Glues consisting of casein, and various oils, bases, and salts. F. G. CROSSE.

XVI.—AGRICULTURE.

Occurrence of red earths and laterites. A. EICHINGER (Z. Pflanz. Düng., 1926, **A8**, 1—13).—The author discusses the origin of red earths and lateritic soils. The enrichment of such soils in sesquioxides is a consequence of the fact that, under moist tropical and sub-tropical conditions, positive ferric hydroxide and aluminium hydroxide sols, arising from the hydrolytic decomposition of silicates, are less stable than silicic acid sols. It is shown by capillary analysis that whilst silicic acid sols can move through capillary spaces, sesquioxide sols are precipitated. The stability of sesquioxide sols in cool moist climates, which leads to the leaching out of the upper soil horizons, may be attributed to the protective effect of humic acids. Tropical soils of the lateritic and red earth type may be classified according to the degree to which lateritisation has proceeded. The most fertile are the younger red earths which still contain undecomposed minerals and are relatively basic, whilst the most infertile are the extreme laterites which are very acid and consist mainly of sesquioxides. G. W. ROBINSON.

Molecular relationships in zeolitic silicates in relation to soil reaction and fertiliser requirements. B. TACKE and T. ARND (Z. Pflanz. Düng., 1926, **A7**, 17—21).—Polemical. The author contests the conclusions of Ganssen (Int. Mitt. Bodenkunde, 1924, **14**, 158) on the significance of the ratio $SiO_2 : Al_2O_3$: bases in the zeolitic silicates of soils. In contradiction of Ganssen's assertion that the reaction of soils with a high ratio of silica to alumina would show a change in reaction after drying, the author gives data to show that no such change in reaction occurs. G. W. ROBINSON.

Determination of replaceable bases in soils. P. S. BURGESS and J. F. BREAZEALE (Science, 1926, **64**, 69—70).—As a displacing liquid, 0·1*N*-barium chloride solution is preferred to ammonium chloride solution. The percolate from 500 g. of air-dried soil is diluted to a definite volume, and filtered under pressure if necessary. Barium is removed from an aliquot part as chromate, the calcium and magnesium (which are not adsorbed) being determined by the soap titration method, or by boiling the phosphates with oxalic acid to precipitate calcium, followed by precipitation of the magnesium as its ammonium phosphate. An aliquot part of the original percolate is employed for the determination of sodium and potassium, Hilgard's methods being applied after evaporation with hydrochloric acid and ammonium sulphate followed by ignition. A. A. ELDRIDGE.

Relation of nitrification processes to the solubility of the phosphoric acid of podsol soil. F. S. SOBOLEV (Nauch. Agron. Zhur., 1925, **2**, 186—198; Chem. Abstr., 1926, **20**, 3327).—There is a parallelism in podsol soils between nitrification and mobilisation of soil phosphate, especially on late fallowed soils, and on both early and late fallowed soils subsequently cropped to rye. The water-soluble phosphate, nitrate, calcium, and hydrogen carbonate contents, and the hydrogen-ion concentration were higher in early than in late fallowed soil, except where cropping to rye followed. The nitrate and phosphate curves of early fallowed soils, the calcium curve of both early and late fallowed soils, and the hydrogen carbonate and hydrogen-ion concentration curves of all soils showed three maxima during the summer, practically simultaneously for the various factors, all occurring during periods of warm and dry weather. In black soils an increase in nitrate content is accompanied by a decrease in the water-soluble phosphate. A. A. ELDRIDGE.

Cultivation sickness in reclaimed peat soils. J. HUDIG and C. MEYER (Z. Pflanz. Düng., 1926, **A8**, 14—52).—A type of cultivation sickness ("Urbarmachungskrankheit") is described which occurs on reclaimed peats in Holland. In soils affected by this disorder the organic matter appears to be in a bad

physical condition, and remains separate from the mineral particles of the soil. The formation of grain and seed on such soils is very greatly depressed. Although some of the soils are very acid, dressings of calcareous material actually depress crop yields. Amongst the ordinary manures and fertilisers, compost containing town refuse is the only dressing which effects an improvement. The effect of town refuse is attributed to the inorganic constituents. Finely crystalline copper sulphate applied at the rate of 50 kg. per hectare causes an improvement in the productivity of " sick " soils similar to that caused by dressings of town refuse. The nature of the " sickness," and the manner in which it is cured by dressings of town refuse or copper sulphate, are as yet unknown. G. W. ROBINSON.

Cell-stimulation and increased yields of crops. GÜNTHER and SEIDEL (Z. Pflanz. Düng., 1926, **A7**, 339—351).—Literature dealing with the stimulation of plant growth by soaking seeds in various salt solutions is reviewed and discussed. Pot experiments are recorded in which a number of stimulating salts are used. Definite increases in yields are reported in some instances, but the results are inconclusive. A. G. POLLARD.

Influence of sulphur and gypsum on the solubility of potassium in soils and on the quantity of this element removed by certain plants. O. M. SHEDD (Soil Sci., 1926, **22**, 325—353).—The addition of sulphur increased the amount of water-soluble potassium in all soils examined. In general, the addition of gypsum did not have this effect. No strict relationship was apparent between the amounts of potassium extracted from soils by solvents (water, nitric acid, and ammonium nitrate solutions were used) and by plants. In most cases, however, increased potassium in the plant followed increased water soluble potassium in the treated soil. Calcium carbonate increased the rate of oxidation of sulphur in soils, but decreased the amount of water-soluble potassium. A. G. POLLARD.

Origin and nature of soil organic matter or soil "humus." IV. Decomposition of the various ingredients of straw and of alfalfa [lucerne] meal by mixed and pure cultures of micro-organisms. S. A. WAKSMAN and F. G. TENNEY (Soil Sci., 1926, **22**, 395—406).—Barley straw, after extraction with ether, was more easily decomposed by bacteria than before; similar results were not apparent in the case of lucerne. Removal of alcohol- and water-soluble matter from lucerne reduced the rate of its decomposition by micro-organisms; the decomposition of straw was unaffected by this treatment. Removal of lignins from both straw and lucerne increased both the rate and extent of decomposition. Lignins remain unaltered in soil for extended periods, and can be recovered by the ordinary methods of extracting " humus." A. G. POLLARD.

Catalase effect in caraway seeds as an indication of their germinating power. E. PETERSOHN (Z. Pflanz. Düng., 1926, **A8**, 99—102).—By means of their catalase reaction with hydrogen peroxide it is possible in the case of many seeds to distinguish good seeds from those of which the germinating power has been destroyed by heating or extraction. In the case of caraway seeds the method is unsatisfactory. By first treating sections of the seeds with a 10% alcoholic solution of guaiacum resin and then placing them in hydrogen peroxide, good seeds produce a blue coloration. By this means, good seeds can be distinguished from seeds which have been extracted or distilled for essential oil. Heated seeds may be distinguished by using *p*-phenylenediamine hydrochloride in the place of the guaiacum solution. The sections used should contain the embryo. Detailed directions are given for carrying out the test. G. W. ROBINSON.

Losses of dry matter during the drying and storage of crops. F. GIESECKE (J. Landw., 1926, **74**, 231—235).—In the laboratory, drying and storage of crops in experimental work is accompanied by losses in dry matter resulting from respiratory changes. The loss increases with the temperature and period of drying, but no relationship could be traced. Partially dried material loses in dry matter content when stored in a closed vessel. The bulk of individual samples of material affects the dry matter loss during the drying process. Milling the material does not affect these processes. A. G. POLLARD.

Chemical and physiological study of maturity in potatoes. C. O. APPLEMAN and E. V. MILLER (J. Agric. Res., 1926, **33**, 569—577).—Investigation of the chemical changes in potatoes during growth and subsequent storage showed that maturing processes continue during storage, so that, at the end of the rest period, immature potatoes suitable for " seed " have about the same composition and respiratory activity as potatoes matured on the plant, if both are stored under the same conditions. Special attention was given to the nitrogenous constituents. The results did not indicate any chemical or physiological basis for the superiority of immature potatoes for " seed." C. T. GIMINGHAM.

Electrodialysis of the colloidal soil material and the exchangeable bases. S. MATTSON (J. Agric. Res., 1926, **33**, 553—567).—The quantities of bases removable by electrodialysis from the colloidal fractions of two soils representative of widely different types of colloidal material were compared with the quantities removable from the same materials by base exchange. The amounts of uni- and bi-valent bases extracted by *N*-ammonium chloride solution or 0·05*N*-hydrochloric acid were almost identical with those obtained by electrodialysis. Treatment of the electrodialysed colloids with a solution of calcium chloride displaces quantities of acid approximately equivalent to the base exchange capacities of the untreated colloids. Electrodialysis may be looked upon as a form of base exchange in which the hydrogen ions of the water are substituted for the uni- and bi-valent cations removed by the electric current. C. T. GIMINGHAM.

Iodometric determination of phosphoric acid in plant products and soils. F. FRODL (Chem.-Ztg., 1926, **50**, 825—827, 839—840, 868—869).—The method previously described (cf. Preisinger and Frodl, B., 1915, 674) is simplified by the use of oxalic acid in place of sulphuric acid. The ammonium phosphomolybdate precipitate (containing 10—15 mg. of P_2O_5) is filtered and washed on a Gooch crucible, treated with a slight excess of sodium hypobromite solution and a few drops of 0·5*N*-sodium hydroxide solution, and, as

soon as evolution of nitrogen ceases, 0·5—0·75 g. of potassium iodide and 50 c.c. of $2N$-oxalic acid are added, and after about 5 min. the liberated iodine is titrated with $0 \cdot 1N$-sodium thiosulphate. Oxalic acid is easily oxidised by sodium hypobromite if the solution of the latter is neutral or slightly acid; in alkaline solution the reaction takes place slowly. It forms a colourless, stable molybdic acid complex.

C. T. GIMINGHAM.

Physics of spray liquids. V. Paraffin-cresols-soap solutions: detergent action of soaps. R. M. WOODMAN (J. Pomology, 1925, **5**, 43—49; cf. B., 1926, 139).—Certain mixtures of cresols (cresylic acid) and soap (fish oil soft soap) with water will dissolve large quantities of paraffin oil; solutions at all concentrations up to 20% can be made. Such solutions are suggested as winter washes for fruit trees. It is argued that soaps containing phenols are more effective as detergent agents than ordinary soaps. C. T. GIMINGHAM.

Tar distillate washes. Their comparative effectiveness, under different conditions, on various pests, and at increasing concentrations. W. GOODWIN, A. M. MASSEE, and R. H. LE PELLEY (J. Pomology, 1926, **5**, 275—286).—Two standard tar distillate washes and two commercial preparations when tested in the field showed very little protective action against tree pests. More favourable results were obtained in laboratory experiments. W. O. KERMACK.

Oxidations and reductions by iron compounds. BORESCH.—See VII.

Preparation of powdery fertiliser from activated sludge. HAUPT.—See XXIII.

Determination of the toxic substance in insecticides. II. Volumetric determination of copper in insecticides containing iron, arsenic, and mercury. BODNAR and TERÉNYI.—See XXIII.

PATENT.

Fertiliser composition. F. W. FREISE, Assr. to AMERICAN CYANAMID CO. (U.S.P. 1,601,954, 5.10.26. Appl., 23.7.23).—Phosphate rock and calcium cyanamide are added to a mixture of crude nitrogenous material and sulphuric acid. H. ROYAL-DAWSON.

XVII.—SUGARS; STARCHES; GUMS.

Carbonatation of sugar-lime solutions. A. H. W. ATEN, P. J. H. VAN GINNEKEN, and F. J. W. ENGELHARD (Rec. trav. chim., 1926, **45**, 753—771).—The activities of hydroxyl ions in solutions containing sucrose and the hydroxides of potassium, lithium, barium, or calcium respectively, have been determined by means of the hydrogen electrode. Sucrose behaves in dilute alkaline solution (up to $0 \cdot 1N$) as a monobasic acid with dissociation constant $14 \cdot 6 \times 10^{-14}$ at 18° (cf. Michaelis and Rona, A., 1913, ii, 379; Madsen, A., 1901, ii, 228). In solutions of a higher concentration of alkali the constant cannot be calculated. The activity of the metal ions in solutions of potassium, lithium, or barium hydroxides of concentrations up to N is but slightly altered by the addition of sucrose, so that the saccharates are largely ionised in solution. Conductivity measurements made on solutions containing sucrose and any one of the four hydroxides specified above are consistent with complete ionisation of the saccharates in dilute solution. The mobility of the saccharate ion in water at 18° is 23. In more concentrated solutions, and especially with calcium hydroxide, low values of conductivity are obtained, which may be due to the presence of undissociated molecules of mono-, di-, or tri-saccharate.

G. M. BENNETT.

Recovery of sucrose from cane-sugar molasses. E. R. WATSON, K. C. MUKERJEE, D. N. GUPTA, and H. S. CHATURVEDI (J. Indian Chem. Soc., 1926, **3**, 229—244). —Battelle's process (cf. B., 1912, 1195; 1915, 916; 1919, 784 A) is applied to the recovery of sucrose from cane-sugar molasses. By diluting the molasses, boiling with lime to destroy invert sugar, precipitating tricalcium sucrate, and recovering the sucrose from this by means of carbon dioxide, an 83·7% recovery of sugar of 70·5% purity is obtained. Washing the calcium compound with hot water leads to large losses of sugar.

G. M. BENNETT.

p_H of carbonatation process in the manufacture of sugar and properties of proteins present. A. H. W. ATEN, J. P. M. VAN GILSE, and P. J. H. VAN GINNEKEN (Rec. trav. chim., 1926, **45**, 792—802; cf. B., 1925, 1014).—The p_H of the first carbonatation in sugar manufacture for the season 1925—6 is found to be 10·8—11·1. whilst the alkalinities vary from 8·7—13. The highest rate of flocculation of the proteins in the liquor is at p_H 10·0, but the volume of the precipitate is at a minimum at the same p_H value as gives the optimum filtration in the carbonatation process. A possible connexion between these p_H values and the isoelectric points of the proteins is discussed. G. M. BENNETT.

Viscosity determinations of dextrins with the Lawaczeck viscosimeter. A. STIRNUS (Z. Spiritusind., 1926, **49**, 331—332).—For the comparison of viscosities of dextrin solutions, a uniform procedure for making the solution must be adopted. The dextrin is either extracted with cold water and filtered, or it is dissolved in hot water. With some samples of yellow dextrins, both methods give practically identical results, but with other white dextrins the viscosity of the solution made by hot extraction is the higher. The viscosity measured at 20° is more than twice that at 50°. F. R. ENNOS.

Evaluation of [water-]paint adhesives. WAGNER. —See XIII.

PATENTS.

Manufacture of [crystallisable] sugar [from cellulosic materials]. E. FÄRBER, Assr. to INTERNATIONAL SUGAR AND ALCOHOL CO., LTD. (U.S.P. 1,599,462, 14.9.26. Appl., 15.6.25. Conv., 26.6.24).— The crude solution, containing 20—30% of sugar, obtained by the action of concentrated hydrochloric acid on cellulosic material such as wood, is neutralised with calcium carbonate, and is treated, below 20°, with finely divided lime or other alkaline-earth oxide. The precipitated sugar-alkaline-earth compound is filtered off, washed with a little water, and the free sugar regenerated by treatment with carbonic or sulphurous acid. The solution of pure sugar thus obtained is rendered crystallisable and fermentable by adjusting its sugar content to about 40%, adding 0·3% of hydrochloric acid, and heating

at 130° under pressure until hydrolysis to simpler sugars is complete. The product is neutralised with sodium carbonate, and evaporated under reduced pressure to a 70—80% syrup, which is freed from sodium chloride. On cooling, pure sugar crystals are obtained.

T. S. WHEELER.

Manufacture of sugars from cellulose-containing materials. R. RÖMER, Assee. of L. PINK (E.P. 240,475, 24.9.25. Conv., 25.9.24).—Wood waste, in the presence of dilute sulphuric acid, is subjected to the action of steam in an open vessel until it is sufficiently softened, then to the action of steam in a closed vessel. The mass is then filter-pressed, and the free acid in the filtrate precipitated by calcium or barium carbonate. After again filtering the liquor, the neutral hexose is recovered from the filtrate by evaporation. The yield of dextrose is from 25—50%, and is dependent upon the quality of the wood used.

F. G. CROSSE.

XVIII.—FERMENTATION INDUSTRIES.

Preservative principles of hops. VII. J. J. H. HASTINGS, F. L. PYMAN, and T. K. WALKER (J. Inst. Brew., 1926, 32, 484—502).—A comparison is made of the following three methods devised for the determination of the relative antiseptic values of hops: (1) The gravimetric method of Ford and Tait, with slight modifications, including Walker's expression $\alpha + \beta/3$ in place of $\alpha + \beta/5$; (2) Chapman's plate method; (3) a new biological method depending on the development of acidity by *Bacterium Delbrückii*. With commercially dried hops, each method gives results which are similar and of a high order of accuracy. Even although the results of the tests are obtained more quickly by the biological methods and especially by Chapman's method, provided the appropriate organisms are available, it is suggested that, in the present state of our knowledge, the gravimetric or chemical method should be adopted, since it is the most readily standardised. In the third method the use was discontinued of *Bacterium X*, which was found to exhibit considerable variation in growth and virility. It was replaced by *Bacterium Delbrückii*, which had formerly been found suitable by Windisch and others in comparing the inhibiting powers of various solutions of humulon.

C. RANKEN.

Fermentation control. H. W. HARMAN and J. H. OLIVER (J. Inst. Brew., 1926, **32**, 503—519).—The degree of attenuation and nature of the fermentation are largely controlled by the character of the wort, which is dependent upon the degree of modification and diastatic activity of the malt. There is no permanent type of primary yeast, from the fermentation point of view, and yeasts derived from different sources soon respond to the character of the wort and quickly lose any inherited properties. Other factors governing fermentation are the alkalinity of the water, the temperatures of conversion and of fermentation, the race and temperature of the pitching yeast, the influence of aeration, and, finally, the effect of change in the nitrogen-content. The salts content, particularly with regard to the potash and phosphate ratio, is of some importance, and although there is an ample supply of salts in a wort for yeast reproduction, it may be that they are not in an assimilable condition. During fermentation the yeast loses mineral matter of constant composition, whilst the mineral matter of the beer remains of fairly constant composition, from which it is deduced that the salts that are being excreted by the yeast must be a relatively alkaline potassium phosphate. Contrary to the view of Baker and Hulton, the authors believe that excessive nitrogen left in the beer may be harmful, and also that there is enough nitrogen in an average malt extract to satisfy the demands of the yeast in respect of reproduction.

C. RANKEN.

Deacidification of wine by warm storage. T. ROETTGEN (Pharm. Zentr., 1926, **67**, 743—745).—Wine of high acidity may be rendered palatable by promoting the growth of bacteria which transform the excess of malic acid into lactic acid. This may be accomplished by storing for a time at 12—15°, by repeated stirring up with the yeast after the main fermentation, and by omitting the sulphuring of the wine.

F. R. ENNOS.

Detection of fruit wine in grape wine. T. RÖTTGEN (Chem.-Ztg., 1926, **50**, 858—859).—17 c.c. of wine are neutralised with sodium hydroxide solution (1 : 1) and treated with 3 c.c. of a mixture of 85 pts. of 5% copper acetate solution and 15 pts. of 10% ammonium hydroxide. With a pure grape wine a slight precipitate of a dirty white colour is formed, leaving the solution with the blue colour of the copper unaltered. Adulteration with 20% or more of fermented apple or pear must gives a brownish precipitate, the colour of the solution changing to green. The method is only applicable to sound white wines, and not to red wines.

F. R. ENNOS.

Production, composition, and evaluation of Marsala wines. A. KICKTON and P. BERG (Z. Unters. Lebensm., 1926, **52**, 175—194).—The processes of manufacture of Marsala wines are discussed. Numerous analyses are recorded of wines from many sources, and comparisons are made between variations in detail of manufacture and the composition of the final article.

A. G. POLLARD.

Determination of tartaric acid in beverages. P. BERG and J. MÜLLER (Z. Unters. Lebensm., 1926, **52**, 259—264).—The official German method (Zentr. deuts. Reich, 1920, **66**, 1621) for determining tartaric acid is satisfactory where little or no free acid is present, and the majority is combined as potassium bitartaric. Where much free acid occurs the method is less trustworthy, unless the acid is partially neutralised. For this purpose potassium acetate or potassium or sodium hydroxide may be used in quantities not exceeding 0·5 equivalent per 1 equivalent of free tartaric acid.

A. G. POLLARD.

Tartaric acid index in wines accidentally abnormal. FONZES-DIACON (Ann. Falsif., 1926, **19**, 462—465).—By studying the development of the "tourne" at different levels in the same vat it is shown that a diseased wine may resemble one which would be regarded as adulterated with water according to the ordinary rules, the sum of the alcohol and the fixed acid being below 12 and the volatile acidity more than 1. With the affected wine, however, there is a considerable diminution in the tartrate present, so that

the tartaric acid index (cf. B., 1926, 104) which in the unaffected wine was greater now becomes less than unity. The importance of the determination of tartrate and potash in detecting apparent or real adulteration of wines is emphasised. F. R. Ennos.

Salicylic acid as a preservative for samples of wine submitted for examination. Fonzes-Diacon and Laforce (Ann. Falsif., 1926, 19, 466).—The addition of salicylic acid to a wine arrests changes in chemical composition apart from the extract which tends to diminish. The germicidal power of the acid gradually diminishes, but is sufficiently prolonged to maintain the wine in its initial condition, except as regards extract and colour, for a considerable period.

F. R. Ennos.

Sulphurous acid and the keeping of white wines in bottle. J. Dubaquié (Ann. Falsif., 1926, 19, 459—462).—Examination of the suspended matter in certain bottled white wines shows the presence of aluminium, iron, and in some cases copper, the aluminium preponderating. The clouding does not occur in wine stored in bottles of glass free from all but traces of aluminium and iron. Its formation when using ordinary glass bottles is ascribed to the action of sulphurous acid in the wine on the glass, yielding sulphites of aluminium and iron, which are precipitated by the pectinous matter. (Cf. B., 1925, 821.) F. R. Ennos.

XIX.—FOODS.

Use of sodium nitrite in the curing of meat. R. H. Kerr, C. T. N. Marsh, W. F. Schroeder, and E. A. Boyer (J. Agric. Res., 1926, 33, 541—551).—In the curing of meat with salt, fixation of colour is ensured by including sodium nitrate in the pickle; nitrites are formed and, by combination with the hæmoglobin of the meat, fix the colour. Experiments under factory conditions show that sodium nitrite, at the rate of $\frac{1}{4}$—1 oz. per 100 lb. of meat, can be successfully substituted for sodium or potassium nitrate. Meat cured in this way contains no more nitrites than meat cured in the customary manner with nitrate. The length of the curing process is reduced, and the products are in no way inferior in quality or wholesomeness to those obtained when sodium nitrate is used. C. T. Gimingham.

Compounds of arsenic in marine crustaceans and shell fish. A. C. Chapman (Analyst, 1926, 51, 548—563).—The proportion of arsenic found in British and Portuguese oysters did not appear to be directly correlated with their general normality, and varied from a maximum of 10 pts. per million for British oysters (as As_2O_3 on the wet substance) to 70 pts. for the Portuguese samples. Escallops showed 36—85 pts., mussels 36—70 for 3 samples of about 12 each, and 100—119 for 3 other samples, cockles 17—40, whelks 12—40, periwinkles 20—40, lobster (boiled, on edible portion) 36—40 for 3 specimens, 110 on the unboiled and 105 on the boiled flesh for one specimen, Dublin Bay prawns 38—100 for edible portion, 20—70 for the internal organs, prawns 36—174, shrimps 17—50, and spiney lobster 20—45. Sixteen samples of sea water taken at points within a radius of about 4 miles of the Nore lightship were found to contain 0·14—1·0 pt. per million of arsenic, whilst clean river water only showed minute traces. The arsenic in the lobster exists in a form not directly amenable to the reducing action of hydrogen, and can practically all be extracted by alcohol and acetone, and was concluded to be present as a more or less complex organic substance or mixture of substances, soluble in water and alcohol; to be possessed of slight toxic properties compared with arsenious oxide, and sufficiently stable to resist the action of hot dilute hydrochloric acid and 5% caustic soda solution. Snails, fresh water crustaceans, and fish only contain traces of arsenic, and Irish moss (on moisture-free material) 10—24 pts. per million. A person, known to be intolerant of shell fish, consumed 1 lb. of lobster containing 0·5 grain of As_2O_3 per lb. of wet edible flesh, and the arsenic in the urine rose from 1/170th of a grain per gal. to an average of $\frac{1}{2}$ grain in 12 hrs., 4/5th of a grain for the following 24 hrs., and 7/10th for the following 12 hrs., and it was found that in its passage through the body the arsenical compound was not broken up sufficiently to allow of its being directly reduced by hydrogen in the Marsh–Berzelius flask. D. G. Hewer.

Presence of lead and other metallic impurities in marine crustaceans and shell fish. A. C. Chapman and H. Linden (Analyst, 1926, 51, 563—564).—Lead was found present (expressed in parts per million on dry material) to the extent of 25·6 and 6·2 in lobster, 20—10 in mussels, 18—7·2 in periwinkles, 17 in crab, 17—5 in whelks, 9·7—1·3 in cockles and 7·5 in prawns; and copper, expressed similarly, 167 in lobster, 130 in crab, and 115 in whelks. Native oysters contained in 3 samples 12, 20, and 400 parts of lead, whilst the average in sea water was 0·4 part per million. D. G. Hewer.

Examination of fish for formaldehyde. A. R. Tankard and D. J. T. Bagnall (Analyst, 1926, 51, 565—567).—Positive results, of the order of 1—2 parts per million, were obtained for formaldehyde in the case of a large number of fish, but these results may not be due to added formaldehyde. Pre-existing trimethylamine (a characteristic constituent of fish) may become oxidised and give positive reactions with the phenylhydrazine test, but oxidation may proceed beyond the formaldehyde stage, with increasing decomposition, when negative results for formaldehyde will be obtained.

D. G. Hewer.

German honeys. J. Fiehe (Z. Unters. Lebensm., 1926, 52, 244—259).—Analyses of numerous samples of honey from a number of different plants are recorded. Chemical characteristics of the various types are discussed. A. G. Pollard.

Distribution of volatile flavour in grapes and grape juices. J. W. Sale and J. B. Wilson (J. Agric. Res., 1926, 33, 301—310).—The content of anthranilic ester (methyl anthranilate) and of total volatile esters is a measure of the intensity of aroma in grapes and grape products. Anthranilic ester is determined by a method based on that of Matthewson (cf. B., 1923, 9 A). The sample is distilled in steam, 25 c.c. of *N*-hydrochloric acid and 2 c.c. of a 2% sodium nitrite solution are added to the distillate, mixed well, and kept for 2 min. 6 c.c. of a saturated solution of hydrazine sulphate are then added and thoroughly mixed for 1 min.

The liquid is kept in motion while 5 c.c. of a 5% solution of sodium α-naphthol-2-sulphonate are added, and then immediately 15 c.c. of a 25% solution of sodium carbonate. If anthranilic ester is present, a colour, ranging from light pink to deep red, is produced due to the formation of *o*-carbomethoxyphenylazo-α-naphthol-sulphonic acid. The intensity of the colour is a measure of the amount of ester present, which is determined by comparison with standards. The volatile flavour of grapes occurs in the pulp as well as in the skins. Anthranilic ester tends to disappear from grape juice which is stored. C. T. Gimingham.

Sulphur dioxide as a preservative for fruit. B. T. P. Barker and O. Grove (J. Pomology, 1925, **5**, 50—60).—The preservation of fruit pulp (for jam-making) in wooden casks for any considerable time by means of sulphur dioxide is open to the objection that relatively large quantities of the preservative are necessary. As the result of a study of the amount of sulphur dioxide required to preserve whole fruit under various conditions of storage, of the effect on the fruit itself, and of the uses, other than jam-making, to which such fruit could be put, an alternative method to pulping is proposed. Fresh whole fruit can be satisfactorily preserved in sealed vessels in an aqueous solution of sulphur dioxide containing 0·08—0·1%. Absorption by the fruit occurs, and the concentration falls in a short time to about 0·04%, but the growth of micro-organisms continues to be inhibited. The quality of jam made from fruit so preserved is much superior to that made from pulp, and it can be used in other ways, as canned or bottled fruit is used. The cost of preservation is reduced by eliminating the boiling and pulping process. C. T. Gimingham.

Detection of fruit wine in grape wine. Röttgen. —See XVIII.

Patents.

Manufacture of bread. J. Takamine, J. Takamine, jun., and N. Fujita (U.S.P. 1,599,930, 14.9.26. Appl., 25.3.22).—An aqueous extract containing diastatic and proteolytic enzymes, obtained from a vegetable fungus growth, is added to glucose syrup, which serves as a stabiliser. This product increases the fermenting activities of yeast, permitting a reduction in the amount of yeast and sugar, and also in the time required for bread-making. F. R. Ennos.

Producing improved cream for butter making and improved milk for cheese making. F. L. Armitage (E.P. 237,899, 22.7.25. Conv., 2.8.24).—The milk or cream is pasteurised and deodorised by boiling and subsequent cooling in a partial vacuum. The boiling temperature is adjusted, by varying the pressure, to that considered necessary for pasteurisation, and the period of boiling is made sufficient to ensure complete removal of objectionable odours. All operations are carried out in the same container, the heating and cooling being effected by external means. F. R. Ennos.

Pasteurisation, sterilisation, or similar heat treatment of milk and other liquids suitable for food. T. C. Manchester (E.P. 259,821, 8.3.26).—The liquid and its container are exhausted of air, the liquid being simultaneously agitated. During the removal of the air or on its completion an inert non-oxidising gas, *e.g.*, carbon dioxide, is introduced, and the pasteurisation, sterilisation, or similar heating process is then carried out. In the absence of air the vitamin content is preserved during the heating. F. R. Ennos.

Cream-treating process. R. K. Cooney, Assr. to Campbell-Cooney Patents Co. (U.S.P. 1,599,650, 14.9.26. Appl., 28.9.25).—Cream of high acidity is neutralised while cold with a solution of soda ash. It is then pasteurised, and while at the pasteurising temperature the solids are removed by a centrifugal clarifier and the liquid formed by the neutralising solution is separated by a centrifugal separator. The percentage of butter fat is finally reduced to the desired amount by the addition of fresh milk. F. R. Ennos.

Atomisation and desiccation of liquids [milk]. Nestle and Anglo-Swiss Condensed Milk Co. (E.P. 256,190, 15.6.26. Conv., 29.7.25).—Milk and other liquids susceptible to decomposition by heat are dried by atomising a jet of the liquid with a core jet of air or gas of high temperature and velocity, and surrounding the hot air and mist of atomised liquid with a current of air of low temperature and velocity, the rapid and uniform cooling of the product preventing overheating. W. G. Carey.

Apparatus for drying liquids [foods]. F. H. Douthitt (E.P. 260,453, 18.1.26).—Materials such as milk, eggs, malt extract, or fruit juices are dried in a chamber provided with means for spraying liquid and with nozzles for creating a cyclonic current of heated air which travels downwards and inwards from near the top of the chamber. In the lower portion of the drying chamber a zone of cool dry air is produced, into which the desiccated material falls. Vanes rotated by the current of air actuate arms having chains attached which rub on the walls and prevent deposition of material. Cold air is blown into the material as it leaves the drying chamber to prevent depreciation during storage, and a pulveriser is connected to the discharge opening to break up the larger masses. Spray and finely-divided material carried away by the hot air are recovered and returned to the drying chamber. W. G. Carey.

Digesting and concentrating protein and preparing non-alcoholic drug extracts. E. Monti (U.S.P. 1,602,163, 5.10.26. Appl., 27.5.21).—The proteins of egg, milk, blood, etc. are digested in grape-juice etc.; the product is dissolved in a fruit or berry juice of higher acidity, and concentrated at a temperature not above 40°. B. Fullman.

Flavouring compound [sodium glutamate]. P. N. Woo (U.S.P. 1,602,958, 12.10.26. Appl., 15.4.26).—Wheat gluten (20 pts.) is heated with concentrated hydrochloric acid (15 pts.) at 65° until solution is complete, about 0·5% of granulated tin is added, and the mixture is heated in an autoclave, under a pressure of 2 atm., for 6—8 hrs. The solution is filtered, concentrated to half bulk, and cooled. The crude glutamic acid hydrochloride, which separates, is neutralised with aqueous sodium hydroxide, and the solution obtained is, after filtration, concentrated to *d* 1·30, filtered from sodium and ammonium chlorides, and treated

with 10 times its volume of alcohol to separate sodium glutamate, which is of value as a flavouring compound for soups etc. T. S. Wheeler.

Pomace extract. E. Monti (U.S.P. 1,602,162, 5.10.26. Appl., 27.5.21).—Fruit pomace extract, from which the pectin and other colloids have been removed, is concentrated to d 1·25 and mixed with whole fruit pomace extract concentrated to d 1·4. The product has a sp. gr. of about 1·3, and contains less than 50% of the sugar, pectins, and other colloids of the raw fruit, but nearly all of the non-sugar, crystalloidal extract of the fruit. B. Fullman.

Method of preserving fodder. A. Messmer, Assr. to M. Flubacher-Brodbeck (U.S.P. 1,603,136, 12.10.26. Appl., 16.12.24. Conv., 4.2.24).—To preserve green fodder, the fresh-cut plant, which has not been acted upon by dew or rain, is placed in an air-tight container, and uniformly sprayed with a solution containing sodium chloride, calcium chloride, sodium phosphate, and ferrous lactate, to sterilise the butyric ferment. B. Fullman.

Ice manufacture [for preserving fish]. M. Oshima (E.P. 260,194, 26.6.26).—Ice for preserving marine fishes is made from sea water to which hydrogen peroxide has been added. Agitation of the mixture during freezing is avoided. H. Holmes.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Properties of glycol dinitrate. H. Rinkenbach (Ind. Eng. Chem., 1926, **18**, 1195—1197).—The principal physical properties of glycol dinitrate have been determined and found to offer certain advantages, such as lower f.p., lower sensitivity to shock, greater fluidity, and perfect oxygen balance, and no serious disadvantages in comparison with those of glyceryl trinitrate as a constituent of explosives. At ordinary temperatures glycol dinitrate is completely miscible with ether, benzene, toluene, acetone, carbon tetrachloride, chloroform, bromoform, aniline, nitrobenzene, glacial acetic acid, furfuraldehyde, glycol diacetate, glyceryl trinitrate, and methyl alcohol. It is immiscible with or only slightly soluble in carbon disulphide, glycol, and glycerol. It is more soluble in water than glyceryl trinitrate, but is practically non-hygroscopic. It has a low vapour pressure, which is, however, approximately 150 times as great as that of glyceryl trinitrate. Rather wide variations were obtained in the determination of the f.p. of glycol dinitrate, indicating the possible existence of isomeric forms of approximately the same f.p., similar to those found in the cases of glyceryl trinitrate. The distinct difference in the refractive indices presents a possible method of analysis for mixtures of the two nitrates. Pure glycol dinitrate has d^{0}_{15} 1·5176, d^{15}_{15} 1·4962, d^{25}_{15} 1·4817; f.p. $-22·3°$; molecular depression of f.p., 41·7; heat of fusion, 30·0 g.-cal./g.; heat of combustion at constant volume 1763·9 g.-cal./g., 268·22 kg.-cal./mol., at constant pressure 1752·5 g.-cal./g., 266·48 kg.-cal./mol.; n^{0}_{D} 1·4546, n^{15}_{D} 1·4491, n^{20}_{D} 1·4436, $n^{22·3}$ (white light), 1·4452; heat of formation (calc.) 365·5 g.-cal./g., 55,580 g.-cal./mol., $\eta^{23·6}$ 0·0363, $\phi^{23·6}$ 27·54; vapour pressure at 0° 0·007 mm. Hg, at 22° 0·0565 mm. Glycol diacetate has $\eta^{23·6}$ 0·0253, $\phi^{23·6}$ 39·52. The following constants were also determined for glyceryl trinitrate: $\eta^{23·6}$ 0·288, $\phi^{23·6}$ 3·47, $n^{22·3}$ (white light) 1·4713; heat of combustion at constant volume 1630·4 g.-cal./g., 370·25 kg.-cal./mol., at constant pressure 1622·1 g.-cal/g., 368·36 kg.-cal./mol.; heat of formation (calc.), 359·8 g.-cal./g., 81,700 g.-cal./mol. Sensitivity tests gave for the minimum fall of a 500-g. weight causing explosion, for glycol dinitrate 110 cm., for glyceryl trinitrate 70 cm., for glyceryl trinitrate containing polyglyceryl nitrates 90 cm. The physiological action of glycol dinitrate is similar to that of glyceryl trinitrate, but owing to its higher vapour pressure the inhalation of sufficient vapour to cause unpleasant effects is more likely to occur during handling. W. J. Powell.

Detection of salicylic acid in presence of digallic acid. N. Bordin (Boll. Chim. farm., 1926, **65**, 642—645).—The presence of phenol in salicylic acid may be detected by treating the substance with sodium carbonate solution and ether, evaporating the ethereal liquid, and testing the residue by the ordinary reactions for phenol. Chlorides may be detected by treating with silver nitrate solution a solution of the acid in water acidified with nitric acid. If, in a test-tube previously rinsed with sulphuric acid, a few dg. of salicylic acid give a yellow coloration with a few drops of concentrated sulphuric acid, foreign organic substances are present. In presence of digallic acid, salicylic acid or a soluble salicylate may be detected by precipitating the former acid as albumin tannate and testing the filtered solution with dilute ferric chloride solution. T. H. Pope.

Determination of phosphorus in oils. W. P. H. van den Driessen Mareeuw (Pharm. Weekblad, 1926, **63**, 1309—1320).—The present methods of determining phosphorus in pharmaceutical phosphorus oils are considered, and the following rapid method suggested. The oil is dissolved in absolute alcohol, and titrated with alcoholic potassium hydroxide, using phenolphthalein, and a comparison colour solution to determine the end point; this gives the original acidity + phosphorous acid formed by oxidation during keeping. Excess of potassium hydroxide and hydrogen peroxide are added, and the excess of alkali determined by titration after keeping 24 hours. The difference of the two titrations gives the phosphorus oxidised by the peroxide, and the original acidity of the oil being known or determined in a phosphorus-free sample, the total phosphorus can be calculated. Oils containing phosphorus may be preserved for many months unchanged by addition of 5% of alcohol and ether, and 1% of oil of lemon. S. I. Levy.

Volatile oils from *Xanthorrhœa arborea*, *X. hastilis*, and *X. reflexa*. H. H. Finlayson (J.C.S., 1926, 2763—2767).—The volatile oils from the above-named resins were isolated by the distillation of an alkaline solution of the resin with steam. The yields were (*a*) *X. arborea*, 1%; (*b*) *X. hastilis*, 1·56%; (*c*) *X. reflexa*, 6·86%. Examination of the phenolic and non-phenolic constituents gave the following results: (*a*) pæonol, 50%; hydroxypæonol, 6·5%; non-phenolic substances, b.p. 115—190°/18 mm., including the odoriferous constituents, 17%; and resinous substances probably formed by oxidation and polymerisation

of other constituents of the oil. (*b*) Phenols, 5%; cinnamyl alcohol, 53%; low-boiling alcohols (probably mostly citronellol), 8%. (*c*) Pæonol, 62·8%; hydroxypæonol, 2·9%; other phenols, 1·5%; benzaldehyde, 3·2%; an aromatic ketone containing one *p*-methoxyphenyl group (since on oxidation anisic acid was obtained), 3%; and an optically active compound, probably $C_{14}H_{14}O_2$, m.p. 68—69°, previously isolated from *X. Preissii* (cf. B., 1920, 459 A), 17·5%.

H. Burton.

Essential oil of *Zieria macrophylla* (Bonpland) and the presence of a new cyclic ketone. A. R. Penfold (J. Proc. Roy. Soc. N.S.W., 1926, **60**, 104—112).—Distillation of the leaves and terminal branchlets of *Z. macrophylla* from Tasmania yields 0·28—0·66% of a reddish-brown oil of unpleasant odour, having d_{15}^{15} 0·9465—0·9704, $[\alpha]_D^{20}$ —39·0° to —66·4°, n_D^{20} 1·5015—1·5148, ester value 29·4—52·8, ester value after acetylation 50·9—71·6, solubility in 80% alcohol (by wt.) 1 in 0·8 to 1 in 1·1 vols., and containing *d*-limonene (10—20%), a cyclic ketone *zierone*, $C_{13}H_{20}O$ (50—60%), a sesquiterpene, a sesquiterpene alcohol, amyl alcohol (?), a low-boiling *iso*valeric ester, unidentified phenolic compounds, a paraffin, m.p. 56° and formic acid. Zierone (*semicarbazone*, m.p. 180—181°, $[\alpha]_D^{20}$ —137°; *phenylhydrazone*, m.p. 107—108° (decomp.), $[\alpha]_D^{20}$ —279·5°) is a pale yellow liquid with an odour of fresh cedarwood, and has b.p. 142·5—144°/10 mm., d_{15}^{15} 0·9752, $[\alpha]_D$ —141·2°, n_D^{20} 1·5140—1·5144, [R] 59·53. It appears to belong to the irone-ionone series.

E. H. Sharples.

Essential oils of *Leptospermum lanigerum* (Smith). I. A. R. Penfold (J. Proc. Roy. Soc. N.S.W., 1926, **60**, 73—84).—Oils from two forms of *L. lanigerum*, *i.e.*, "green" leaf (form "A") and "silver" leaf, which are regarded as being extreme forms of one another, are described. The leaves and terminal branchlets were steam-distilled in each case. "Silver" leaf yielded 0·28—0·33% of oil having d_{15}^{15} 0·9152—0·9231, $[\alpha]_D^{20}$ nil to + 10·5°, n_D^{20} 1·4890—1·4928, ester value 12·81—29·63, ester value after acetylation 62·59—100·08, and containing sesquiterpenes (aromadendrene and eudesmene), 60—75%, *d*-α-pinene, 16—20%, with small quantities of a sesquiterpene alcohol, *iso*valeric ester, geraniol, geranyl formate and cinnamate, citral, cineole, and unidentified phenolic substances. "Green" leaf yielded 0·46—0·67% of oil having d_{15}^{15} 0·9047—0·9242, $[\alpha]_D^{20}$ + 30·1° to + 32·2°, n_D^{20} 1·4756—1·4783, ester value 42·67—56·89, ester value after acetylation 92·15—150·32, and consisting of *d*-α-pinene, 40—60%, darwinol (naphthylcarbimide, m.p. 90—91°) and its acetic ester, 40—45%, a sesquiterpene, a sesquiterpene alcohol and esters, and unidentified phenolic substances.

E. H. Sharples.

Essential oils from some cultivated eucalypts. I. A. R. Penfold (J. Proc. Roy. Soc. N.S.W., 1926, **60**, 55—59).—The oils described were obtained by steam-distillation of the leaves and terminal branchlets from trees grown in good garden soil and having access to a moderate quantity of moisture. *Eucalyptus Australiana* yields 2·4—2·6% of oil (d_{15}^{15} 0·9221—0·9223, $[\alpha]_D^{20}$ + 2·5° to + 3·0°, n_D^{20} 1·4634—1·4640, solubility in 70% alcohol, 1 in 1·1 vols.) containing 56—60% of cineole and no phellandrene. *E. Macarthuri* yields 0·5—0·74% of oil (d_{15}^{15} 0·9257—0·9356, $[\alpha]_D^{20}$ + 3·5° to + 4·8°, n_D^{20} 1·4696—1·4771, solubility in 70% alcohol, 1 in 1·2 to 1 in 1·3 vols.), containing geranyl acetate, 61·9—70·2%, geraniol, 3—6%, and eudesmol, 16·2—25·0%. *E. radiata* (*numerosa* Maiden) yields 2·7% of oil (d_{15}^{15} 0·8884, $[\alpha]_D^{20}$ — 55·4°, n_D^{20} 1·4771, solubility in 80% alcohol, 1 in 0·6 vol.), containing 20% of piperitol and 19·5% of piperityl ester. *E. citriodora* yields 0·5—1·0% of oil (d_{15}^{15} 0·8607—0·8705, $[\alpha]_D^{20}$ — 1·1° to nil, n_D^{20} 1·4498—1·4558, solubility in 70% alcohol 1 in 1·2 to 1 in 1·3 vols.), containing 90—98% of citronellal. These oils are superior in colour and aroma to the usual commercial samples.

E. H. Sharples.

Isolation and identification of certain thiophen compounds occurring in shale oil. Scheibler.—See II.

Patents.

Preparation of butyric acid. C. O. Young, Assr. to Carbide and Carbon Chemicals Corp. (U.S.P. 1,599,737, 13.10.22).—Butaldehyde is passed into a reaction chamber containing an oxidising atmosphere maintained at a temperature above the boiling point of the aldehyde at the pressure prevailing in the space, but low enough to cause a liquid containing butyric acid to be formed. The liquid is collected at a point remote from that at which the aldehyde is introduced and the butyric acid recovered from it by distillation.

A. Davidson.

Manufacture of methyl alcohol. S. P. Burke, Assr. to R. H. McKee (U.S.P. 1,602,846, 12.10.26. Appl., 7.9.22).—Methyl ether is converted into methyl alcohol by means of steam at 350—375° in the presence of an agent having the essential properties of aluminium oxide.

A. J. Hall.

Manufacturing ketens. D. A. Nightingale, Assr. to Ketoid Co. (U.S.P. 1,602,699, 12.10.26. Appl., 22.7.24).—Acetic anhydride or acetone, passed, at 635°, over chromium, manganese, aluminium, calcium, barium, strontium, magnesium, or silver sulphate, or a mixture of these sulphates, yields keten in 80% yield.

T. S. Wheeler.

Nitrogenous derivatives of terpene alcohols. R. Wolffenstein (E.P. 260,159, 24.3.26).—Esters of terpene alcohols are prepared by treatment with pyridinecarboxylic acids in presence of a condensing agent, or by treatment of the alcohols or alcoholates with the acid halides. The products form well crystallised salts which serve in place of the usual distillation for their purification. *E.g.*, 41 g. of nicotinic acid and 50 g. of menthol are mixed in 100 g. of benzene, phosphorus oxychloride is added, and the mixture boiled under reflux for some time. After cooling, sodium carbonate solution is added till alkaline, the benzene solution separated, and the benzene removed. The residue is dissolved in ether and an ethereal solution of sulphuric acid added, when *menthyl nicotinate sulphate*, m.p. 156—157°, crystallises out. *Bornyl nicotinate sulphate*, m.p. 135°, and *bornyl picolinate sulphate*, m.p. 140°, are prepared in a similar manner. By heating picolinyl chloride with menthol in xylene at 150—160°, *menthyl picolinate* is formed. The esters find therapeutic application.

A Davidson.

Production of 5-nitro-2-hydrazinopyridine. Deutsche Gold & Silber-Scheideanstalt (E.P. 255,811, 12.2.26. Conv., 22.7.25).—A 2-halogeno-5-nitropyridine is acted on by hydrazine hydrate or an aqueous solution of it in the cold. *E.g.*, 10 g. of finely-powdered 2-chloro-5-nitropyridine are mixed with 13 g. of a 50% aqueous solution of hydrazine, and the mixture kept, with occasional stirring, for about 12 hours. The paste is then diluted with water and the 5-nitro-2-hydrazinopyridine, decomp. 204°, filtered off. If pure hydrazine hydrate is used instead of the aqueous solution the reaction takes place at once. A. Davidson.

Manufacturing nitriles of the perylene series. F. Bensa, Assee. of A. Pongratz (E.P. 254,310, 22.6.26. Conv., 26.6.25).—Halogenoperylenes are heated with cyanides at high temperatures with or without solvents. *E.g.*, a mixture of 1 pt. of dichloroperylene (E.P. 244,739, B., 1926, 974) with 1 pt. of cuprous cyanide is heated to about 300°. A mobile liquid forms at first, but this thickens later to a stiff paste. After cooling, the finely-powdered product is extracted with aqueous ammonia, then dissolved in hot nitrobenzene, filtered from undissolved residue and cooled, when the *dicyanoperylene*, brownish-red, crystallises. If the mixture of dichloroperylene and cuprous cyanide is boiled with quinoline, a *chloroperylenenitrile*, m.p. 316°, is formed. A. Davidson.

Preparation of an anthelmintic. Farbenfabr. vorm. F. Bayer & Co. (E.P. 243,325, 27.10.25. Conv., 21.11.24).—Anthelmintics are prepared by extraction of the caoutchouc from the uncoagulated or coagulated latex of species of *Ficus*. For example, 1000 pts. of the uncoagulated latex of *Ficus glabrata* are mixed with 2000 pts. of acetone and, after separation from the supernatant liquid, the residual tough mass is extracted with light petroleum, the temperature being kept below 30°. E. H. Sharples.

Manufacture of pharmaceutical products. Sir W. J. Pope (E.P. 260,346, 28.7.25).—By reaction between boric acid and anæsthetic alkaloids or synthetic bases, or between barium borate and the sulphates of the alkaloids or bases, complex boric acid derivatives of the latter are formed, in which 1 mol. of base is associated with 5 atoms of boron (pentaborates). These pentaborates have a greater anæsthetic effect than the salts of the bases commonly used, this being ascribed to their p_H value, which is about 8 when they are diluted with 50 volumes of water. The following *borates* are described: borate of ethocaine, $2\,C_{13}H_{20}N_2O_2,5B_2O_3,4H_2O$, m.p. 168°; of butyn, $C_{18}H_{30}N_2O_2,5HBO_2$, m.p. 197°; and of amydricaine, benzamine, cocaine, amylocaine, glycocaine, benzocaine, and phenocaine. B. Fullman.

Manufacture of solutions of aminoaryldichloroarsines or aminoarylarsenious oxides. G. Newbery, and May & Baker, Ltd. (E.P. 260,382, 29.8.25).—If solutions of aminoaryldichloroarsine hydrochlorides or their substituted derivatives are treated with a limited quantity of an organic base, such as piperazine or ethylamine, a relatively stable solution of the free aminoaryldichloroarsine is obtained. Use of a larger quantity of base gives neutral or alkaline solutions of lower stability (but which can be preserved in absence of oxygen), containing aminoarylarsenious oxides. These various solutions are of therapeutic importance. Details are given of the preparation of 3-amino-4-hydroxyphenyldichloroarsine and of 3-amino-4-hydroxyphenylarsenious oxide from the corresponding arsine hydrochloride. B. Fullman.

Reducing nitro-compounds (E.P. 260,186).—See IV.

Drug extracts (U.S.P. 1,602,163).—See XIX.

Sodium glutamate (U.S.P. 1,602,958).—See XIX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Patents.

Photographic emulsion with mercury compound. S. E. Sheppard and J. H. Hudson, Assrs. to Eastman Kodak Co. (U.S.P. 1,602,589, 12.10.26. Appl., 4.11.25).—The emulsion consists of a colloid with sensitive mercury salt in suspension, and an added compound upon which part at least of the light-sensitiveness of the emulsion depends. This compound contains a bivalent atom of the sulphur group directly linked by a double linking to a single metalloid atom, to which another group of atoms is attached. The proportion of this compound is independent of that of the colloid. W. Clark.

Fluorescent material. S. E. Sheppard, Assr. to Eastman Kodak Co. (U.S.P. 1,602,593—4, 12.10.26. Appl., 17.2.26).—The fluorescent material is incorporated with a tungstate having high fluorescent properties when excited by *X*-rays, and a compound of (a) vanadium or (b) molybdenum which is capable, under oxidising conditions, of insuring the presence of (a) vanadic or (b) molybdic acid. W. Clark.

***X*-Ray protective material.** W. G. Lindsay, Assr. to The Celluloid Co. (U.S.P. 1,602,688, 12.10.26. Appl., 6.10.22).—The material consists of nitrocellulose, tricresyl phosphate, and a substance impervious to *X*-rays diffused through the mass in sufficient quantity to protect efficiently against the injurious effects of the rays. W. Clark.

XXII.—EXPLOSIVES; MATCHES.

Testing the stability of nitrocellulose and smokeless powder by N. L. Hansen's hydrogen-ion method. L. Metz (Z. ges. Schiess- u. Sprengstoffw., 1926, **21**, 186—188).—Hansen has proposed to measure the stability of nitrocellulose and smokeless powder by determining the concentration of hydrogen ions in the aqueous extract of the sample after it has been heated at 110° for a known time. During the heating nitric oxide is evolved, and is oxidised to nitrogen peroxide which forms, with moisture, nitrous and nitric acids. With further denitrification weak acids such as acetic, oxalic, and mucic acids are formed which pass into the aqueous extract. The concentration of the hydrogen-ions in the extract is then determined and the value thus obtained is a measure of the extent to which the nitrocellulose or powder has decomposed. The hydrogen-ion concentration is determined at 18—20° by the quinhydrone electrode method and the p_H values so obtained are calculated to values for $C_H \times 10^5$. A

curve showing the rate of increase of $C_H \times 10^5$ is obtained by heating samples at 110° and withdrawing one every hour, extracting it with water, and determining the p_H. From the slope of the curve obtained by plotting the values for $C_H \times 10^5$ as ordinates against the times of heating as abscissæ, the stability of the nitrocellulose or smokeless powder can be deduced. Some curves obtained by testing various guncottons in this way are given. S. BINNING.

The pressure wave sent out by an explosive. II. W. PAYMAN and W. C. F. SHEPHERD (Safety in Mines Res. Bd., 1926, No. 29, 28 pp.).—The rate of propagation of the shock and pressure waves sent out by an explosive, and the rate of projection of the flame and detonation products have been investigated, photographically, by the wave-speed camera. The atmospheric temperature directly affects the rate of propagation of the spherical shock wave, this rate being about 600 m./sec. in a free space for the first 30 cm. of travel, using a No. 6 detonator (as used for coal mining operations). The plane wave confined in an experimental tube travels at 1150 m./sec. At 6 m. from the mouth of the tube the speed of the shock wave has fallen to 360 m./sec., but the detonation products cease to move forward when about 1 m. from the tube. The shock wave has been observed to travel more slowly than sound. The flame of the detonation is "blanketed" by non-combustible gases, and cannot, therefore, ignite a mixture of air and firedamp. R. A. A. TAYLOR.

Glycol dinitrate. RINKENBACH.—See XX.

XXIII.—SANITATION; WATER PURIFICATION.

Preparation of powdery fertiliser from activated sludge in Milwaukee. H. HAUPT (Z. angew. Chem., 1926, **39**, 1402—1406).—In this process, which has been in operation on the large scale for 12 months, 10% of the sludge deposited, containing 98% of water, is withdrawn for treatment, the remainder being returned to the sewage purification plant. To this sludge aluminium sulphate and sulphuric acid are added with air agitation until $p_H = 3 \cdot 37$, and the acidified sludge is heated by means of heat exchangers and exhaust steam to 72—80°. The sludge, in quantity 4500—10,000 cub. m./day, is then passed by cast-iron pipes to rotary Oliver filters, presses and centrifugals having been found to be unsuitable. These filters revolve in 8—15 min., operate under a vacuum of 2·8 m. of water in summer and 5·6 m. in winter, and the wood and cloth are mechanically brushed and sprayed clean. The sludge, still containing 80% of moisture, is conveyed to Atlas rotary driers, where in 40 min. the moisture content is reduced to 10%. The dried material is severed, and that part which passes a 2·5 cm. mesh is ground and stored. The larger portions are returned to the drier with the wet material. An analysis of the product shows a content of 2·34% of total phosphoric acid and 7·32% of nitrogen as ammonia, of which latter 63% is available. C. IRWIN.

Determination of the toxic substance in insecticides. II. Volumetric determination of copper in insecticides containing iron, arsenic, and mercury. J. BODNAR and A. TERÉNYI (Z. anal. chem., 1926, **69**, 260—282).—The substance is dissolved in water, dilute sulphuric acid, or nitric and sulphuric acids according to its composition; organic matter is destroyed by evaporation with strong sulphuric acid. An aliquot part of the solution containing 50—100 mg. of copper is then treated with 10—20 c.c. of 20% sulphuric acid if free from iron, arsenic, and mercury, and the copper determined by the method of Bruhns (B., 1918, 445 A). If iron is present, the solution is treated with solid sodium pyrophosphate until the precipitate first formed just redissolves, then with 20% sulphuric acid until the acidity corresponds with 2—4% sulphuric acid; alternatively, sodium phosphate is added to produce a precipitate, which is then dissolved in phosphoric acid. In both cases the copper is determined by Bruhns' method. If the substance contains arsenic, a little ferric chloride is added previous to the pyrophosphate. In the presence of mercury ammonia is added in excess, and the precipitate filtered off, dissolved in nitric acid, and reprecipitated. The combined filtrates are boiled with 3—5 c.c. of hydroxylamine hydrochloride to precipitate the mercury, and the filtered solution is treated as above. (Cf. B., 1926, 997.) A. R. POWELL.

Spray liquids. V. WOODMAN.—See XVI.

PATENTS.

Permanently preserving animals and plants. F. HOCHSTETTER and G. SCHMEIDEL (U.S.P. 1,602,489, 12.10.26. Appl., 17.12.25. Conv., 19.11.24).—The plant or animal is soaked in a fixing fluid, prepared from glacial acetic acid (1 pt.), absolute alcohol (6 pts.), and chloroform (3 pts.), and, if necessary, the fluid is injected into the veins. Water is then removed by treatment with absolute alcohol containing 3% by vol. of phenol. The alcohol is displaced with benzene containing 3% by vol. of phenol, and, finally, the specimen is treated at 48° with molten paraffin of m.p. about 45°. All excess of paraffin is allowed to drain away, and after that remaining has set the specimen is coated with varnish. T. S. WHEELER.

Circulating and aerating sewage. R. AMES, J. BOLTON, and M. W. MILLS (E.P. 258,102, 22.10.25).—A portion of the mixture of activated sludge and sewage thrown out from a central uptake tube by a rotating cone is caught by open topped dishes or annular trough lying adjacent to the central tube and conveyed by pipes to four-corner settlement pockets formed by partitioning off the four corners of a truncated inverted pyramid-shaped aeration tank. When the quantity of liquid collected by the dishes is in excess of that passing over the outlet weirs of the settlement tanks, a positive flow is created through apertures leading from the base of the settlement pockets to the central aerating tank which keeps the pockets free from settled sludge. W. T. LOCKETT.

Rendering harmless substances which cause hardness in soaping and washing operations. G. ULLMANN (E.P. 259,437, 11.1.26).—A mixture of ordinary soap and a product obtained by saponifying sulphonated fats, oils, resins, or fatty or resinous acids, is added, the latter in an amount less than that equivalent to the substances which cause the hardness. To the mixture may also be added stabilising or peptising agents, *e.g.*, urea, amino-acids, soluble lactates, phosphates, borates, silicates, etc., in small quantities. W. T. LOCKETT.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JANUARY 21, 1927.

I.—GENERAL; PLANT; MACHINERY.

Effect of acids on the mechanical strength of timber. E. A. Alliott (J.S.C.I., 1926, **45**, 463—466 T).—The tests were made mainly on pitch pine, teak, and American oak. Strips of ½ in. cross-section and 7 in. span were broken to determine the effect of soaking in water and in sulphuric, acetic, and hydrochloric acids of various strengths for periods up to 130 days. The loss of strength after prolonged wetting is least in the case of teak (7%), and greatest in the case of pitch pine (33%). After soaking in various acids, pitch pine loses a smaller percentage of its strength as compared with its normal strength after wetting with water, though in many cases teak retains the greater actual breaking strength. Teak does not appear to resist hot acids so well as pitch pine. In general, American oak shows up unfavourably for resisting acids. Rough tests with other timbers, such as beech, kauri pine, etc., indicate that they do not approach pitch pine and teak in resistance; lignum vitæ is an exception, but at temperatures above 80° the resinous matter softens, melting at 95°, and the resistance then decreases.

Heat economy of drying with hot gases. W. Graulich (Chem.-Ztg., 1926, **50**, 921—922).—From thermochemical data, the heat economy of drying material by means of hot gases drawn directly from the fire has been calculated (*a*) when the gases are passed straight over the material, and (*b*) when the gases are first used to preheat the material by passing them around the drying container and then backwards over the stream of damp material in such a way that the issuing gases are saturated with water vapour at 80—150°. From the results obtained the efficiency of the second procedure appears to be nearly 20% higher than that of the former. The use of a large excess of air in the furnace results in a greater fuel consumption than the use of little more than the theoretical. It is recommended that the minimum amount of air be used, and that the products of combustion be cooled to 800° immediately after leaving the furnace by addition of the requisite amount of fresh air. A. R. Powell.

Conduction of heat through powders and its dependence on pressure and conductivity of the gaseous phase. J. Aberdeen and T. H. Laby (Proc. Roy. Soc., 1926, **A, 113**, 459—477).—The thermal conductivity of "silox" powder in air, carbon dioxide, and hydrogen over the pressure range 1—760 mm. is expressed by $k = \frac{1}{2}k_0 \log_{10} p/n$, where k_0 is the conductivity, p the pressure of the gas, and n is a constant for each gas. This is not in agreement with Smoluchowski's theory of conduction of heat through powders. The thermal conductivity of silox in a vacuum was determined as $0\cdot7 \times 10^{-5}$ cal. cm.$^{-1}$ sec.$^{-1}$ deg.$^{-1}$. A powder such as silox appears to possess advantages for insulating liquid oxygen vessels. E. S. Hedges.

Patents.

Fusion furnace. A. F. Gordon and W. H. Groves, Assrs. to Western Electric Co. (U.S.P. 1,603,762, 19.10.26. Appl., 27.4.25).—In a furnace for fusing materials, *e.g.*, specimens of refractory clay, the specimens are supported in spaced relationship from the walls of the furnace, in such manner that heat from a combustion chamber below comes in direct and even contact with the material to be fused. C. A. King.

Vertical disc crushers. E. Symons (E.P. 258,471, 25.1.26).—Mechanical improvements to a gyratory disc crusher with a vertical shaft. B. M. Venables.

[Gyratory] crusher. B. A. Mitchell (E.P. 258,875, 22.9.26. Conv., 24.9.25).—A gyratory crusher has the gyrating part supported from the fixed part with adjustment for the distance between the two, and the whole is supported on yielding cushions. The source of power is preferably a self-contained electric motor, and the gyrations are not positively effected, but only induced by a member rotating within the gyrating member being out of balance; the range of the gyrations is, however, positively limited. One of the crushing members may have holes formed in it for the outlet of crushed material. B. M. Venables.

Gyratory crusher. E. B. Symons, Assr. to Symons Bros. Co. (U.S.P. 1,600,780, 21.9.26. Appl., 17.5.24).—A circular opening in the top of a housing, including a gear-case, is provided with a circular closure formed with a bearing surface extending upwards, and lubricating passages are formed within the walls of the housing and the closure. Unequally-spaced pins are interposed between the housing and the closure, one pin being hollow to connect the lubricating passages. H. Holmes.

Grinding, pulverising, or disintegrating mills. W. A. Cloud (E.P. 260,034, 20.7.25).—The apparatus comprises a number of rotating discs carrying pins which intercalate with fixed pins. The discs increase in size in succession, and around each grinding zone and at the back of each disc is a clearance space which is at least as great in volume as the preceding grinding zone. In the clearance space the material separates, the fine being carried forward to the next grinding zone and the coarse dropping back into the preceding grinding zone. The air current for propelling the material may be produced by self-contained fan blades. If desired, the apparatus may work drowned in liquid circulating from and to an overhead tank. B. M. Venables.

Recuperative apparatus. F. A. FAHRENWALD (U.S.P. 1,599,613, 14.9.26. Appl., 28.11.24).—The gas to be preheated or recuperated is caused to flow upwards through conduits located in each of two hollow upright chambers communicating at their upper ends, and means are provided for delivering heated gases to the bottom of one chamber and discharging them from the bottom of the other chamber. H. HOLMES.

Method and apparatus for removing large-sized particles and aggregates from certain finely divided powders and the like. G. GALLIE, B. D. PORRITT, and RESEARCH ASSOC. OF BRIT. RUBBER & TYRE MFRS. (E.P. 260,741, 7.10.25).—Substances such as carbon black are tested for gritty matter by mixing with water in a funnel-shaped vessel in the neck of which is a tensioned screen of the order of 300 meshes/linear inch. Above the screen is a jet, preferably spreading, of water which washes the powder through the screen with sufficient energy to break up aggregates. The apparatus is stated to be also applicable on a commercial scale. (Cf. B., 1926, 935.) B. M. VENABLES.

Drying devices for granular material and the like. G. HILGENBERG (E.P. 260,896, 11.6.26).—A device for drying, *e.g.*, crystalline salts, without damage consists of a number of annular trays supported by a central rotating vertical duct through which hot gases are passed. The crystals are raked gently from tray to tray by fixed deflectors. B. M. VENABLES.

Cyclone separators or dryers. W. R. WOOD (E.P. 260,776, 5.11.25).—A cyclone separator is shaped entirely as an inverted cone (*i.e.*, without a cylindrical portion at the top), and from a tangential inlet at the top the gas stream passes below a helical baffle which makes one complete turn (downwards) round the interior of the cone. An interior inverted cone may be provided leaving an annular space (which the helical baffle may completely fill) for the incoming gas. The inner cone may serve as an outlet, its lower end being shaped as a Venturi tube, with the object of gradually taking the spin out of the gas. B. M. VENABLES.

Centrifugal separators. A. W. EMPSON (E.P. 260,397, 21.9.25).—The centrifuge, which is suitable for the dehydration and purification of oil, is constructed so that the fluid is treated first in the zone of maximum centrifugal force, whereby the coarser impurities and any water are disposed of by centrifugal action. The fluid is then caused to flow inwards through a filter of superposed sheets of paper or other suitable material to eliminate colloidal substances and suspended matter. W. G. CAREY.

Centrifugal separators. E. W. GREEN and H. OGDEN (E.P. 260,071, 15.8.25).—A centrifugal separator for two liquids, which differ only slightly in sp. gr., is constructed so that the actual overflow weirs comprise uninterrupted circular rings at very nearly the same radius, and so that the light liquid is led away from the bowl nearer the axis, and the heavy from the outermost layers, the latter passing through long narrow passages arranged round a conical cap closing the bowl. B. M. VENABLES.

Separating substances from liquids. W. C. GRAHAM, H. S. RUMSEY, and A. U. WETHERBEE, Assrs. to GILCHRIST & Co. (U.S.P. 1,602,014, 5.10.26. Appl., 5.1.25).—Liquid to be clarified is delivered through a central inlet to a separating chamber, and is withdrawn at points radially remote from the inlet. The sediment is removed radially without opposing the flow of the liquid. The scums are removed through the central inlet. H. HOLMES.

Uniformly drying or processing materials. A. E. STACEY, JUN., Assr. to CARRIER ENGINEERING CORP. (U.S.P. 1,599,797, 14.9.26. Appl., 12.8.24).—The material is subjected to successive currents of air or gas of different velocities, and the currents of the highest velocity are caused to impinge on different zones of the material at different times. H. HOLMES.

Apparatus for drying and heating. W. G. GRAVES, Assr. to GRASSELLI CHEMICAL Co. (U.S.P. 1,599,467, 14.9.26. Appl., 31.1.23. Renewed 22.1.26).—A multiple closed-hearth furnace provided with revolving rabbles includes superposed closed muffles beneath and in series with the drying hearth. A passage is arranged through the furnace for preheating a gas out of contact with the material, and means are provided between the passage and the drying hearth for forcing the preheated gas over the material on this hearth and through an outlet from the drying hearth zone. H. HOLMES.

Evaporators for steam power plants. A.-G. BROWN, BOVERI & Co. (E.P. 255,050, 25.6.26. Conv., 9.7.25).—Several evaporators are used for distilling the make-up feed water, each being heated by steam tapped from a different pressure stage of the prime mover, and the vapours produced are condensed by the feed water flowing successively through corresponding separate preheaters. H. HOLMES.

Evaporators. H. HILLIER (E.P. 260,387, 8.9.25).—An evaporator of the marine type has its steam coils divided into two, so that two inlet headers and one outlet may be provided (or *vice versa*) and a lower pressure of steam used. The headers may also be subdivided, so that steam from more than one source may be used. B. M. VENABLES.

Continuous crystallising apparatus. E. C. R. MARKS. From GRASSELLI CHEMICAL Co. (E.P. 260,691, 11.8.25).—Crystallisation takes place while the liquid ascends through the space between two inverted cones. Both or either of the walls of the crystallising space may be water-cooled. Below the crystallising space is another inverted conical vessel in which the crystals are collected, and to the upper part of which the original liquor is supplied. Above the crystallising space the outer wall is expanded in the form of an enlarged cone, in which are situated heating coils, so that the mother-liquor which overflows from the rim of the enlarged cone will not be saturated. B. M. VENABLES.

Separators for removing entrained oil from hot vapours. J. R. STERLING (E.P. 260,899, 14.6.26).—The separator is of the gravity type, and has hollow baffles inside the separating chamber; each baffle is subdivided by an internal partition into two compartments, and cooling water circulates in succession through the two portions of each baffle. The hot gaseous medium impinges on the cold surface, and the entrained oil is deposited in the form of droplets. W. G. CAREY.

Continuous filtration. F. W. Manning (U.S.P. 1,604,649, 26.10.26. Appl., 28.1.24).—A treating agent is introduced into a chamber containing a liquid, and the treated portion is immediately filtered through the agent, while that portion of the liquid yet to be filtered is treated by mixing the agent with it. W. G. Carey.

Continuous counter-current pressure filtration. F. W. Manning (U.S.P. 1,604,650—1, 26.10.26. Appl., [A] 3.11.24; [B] 2.4.25).—[A] An enclosed chamber is divided into two compartments by a filter wall. The liquid to be filtered is treated with an agent in one portion of the chamber, is filtered through the partition, and is then treated with another agent in the other portion of the chamber. Means are provided for forming the solids into a filter cake, and for maintaining the cake at a constant thickness. [B] A portion of a cylindrical filter wall out of contact with the liquid to be filtered is continuously coated with a treating solid, which by a spiral feed is continuously moved bodily over the wall into contact with the liquid to be treated and filtered; this liquid then passes through the coating of solids and the filter wall. W. G. Carey.

Drying gases and vapours. W. Carpmael. From I.-G. Farbenind. A.-G. (E.P. 260,914, 6.7.26).—A continuous process for drying gases with active material such as silica gel, active alumina, etc. is attained by feeding it through a revolving drum in counter-current to the gas to be dried. The active material, charged with water, is regenerated in a similar drum with direct heating on a counter-current principle, means being provided for feeding material from one drum to the other. W. G. Carey.

Removing gases from boiler feed water by a vacuum. P. Müller, G.m.b.H. (E.P. 254,707, 25.6.26. Conv., 3.7.25).—Sludge or easily-soluble salts are continuously removed from a boiler, and the steam produced from them by the reduction in pressure is passed through an air extractor of the steam-jet ejector type, which communicates with the feed water reservoir. W. G. Carey.

Manufacture of articles having surfaces capable of producing diffraction colour effects. F. Twyman, A. B. Klein, and H. Sayer (E.P. 260,669, 4.8.25).—The substance, such as cellulose acetate, albumin, varnish, etc., is poured on to a suitably heated cylinder of optically polished hardened steel ruled as for a diffraction grating, and the resulting film is removed. W. G. Carey.

Centrifuge. T. C. Thomsen, Assr. to Koefoed, Hauberg, Marstrand, & Helweg, Aktieselskabet Titan (U.S.P. 1,603,726, 19.10.26. Appl., 9.4.25. Conv., 10.4.24).—See E.P. 232,213; B., 1925, 528.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Calorimetric method of determining the calorific value of fuels. W. Steuer (Brennstoff-Chem., 1926, **7**, 357—359).—A historical review of the development of the calorimetric method for fuels from the earliest experiments of Lavoisier and Laplace in 1781 to the present day. W. T. K. Braunholtz.

Working with reducing gases in apparatus made of copper or its alloys. J. Y. Johnson. From I.-G. Farbenind. A.-G. (E.P. 260,888, 2.6.26).—The permeability of copper to hydrogen and other reducing gases at high temperatures and pressures is overcome by fitting the copper vessel or pipe with an inner jacket of copper, so that a narrow space is formed, and filling this space with a gas which will not reduce the oxides of copper or its alloys. The gas may be circulated or may remain quiescent, but preferably it is maintained at the same pressure as the reducing gas. W. G. Carey.

Determination of nitrogen in coal. A. van Steenkiste (Natuurwetensch. Tijdsch., 1926, **8**, 88—91).—Where only occasional determinations are required, the ordinary Kjeldahl method involves the least trouble, but the results are always low, probably by loss of nitrogen as such. Ter Meulen's method, which consists in mixing the powdered coal with barium carbonate, heating to bright redness in a current of hydrogen and steam, and passing the gases over finely-divided nickel at 350°, the ammonia formed being collected in the usual way, is more accurate and much more rapid once the necessary apparatus has been erected; it is, therefore, most suitable where frequent and regular determinations must be made. S. I. Levy.

Small chamber coke oven. A. Schmolke (Stahl u. Eisen, 1926, **46**, 1582—1585).—The performance of a battery of thirty regenerative coke ovens, having chambers 35 cm. wide with conical tops, is examined. Coal is not compressed before coking, and the coke produced is correspondingly more porous than that from coal which has been stamped. It has great strength, with only a slight tendency to crack. By reducing the coking period to 12 hrs. the production of coke is increased, the reduction to coke being simultaneous throughout the whole chamber, hence its superiority over coke produced in a wider type of oven in which the finished coke is exposed to the full temperature of the oven for long periods to complete the coking in other parts of the chamber. With simultaneous completion of the process the gas, otherwise expended unnecessarily and harmfully on the overheated coke, is saved. The thermal economy thus obtained is demonstrated by the small amount of heat required for coking. A large volume of gas is thus produced which is not decomposed, since, at the end of the coking period, gas collected over the mass of coke does not attain the decomposition temperature. The whole of the ammonia, benzol, and tar may, therefore, be recovered, and the high yield of by-products further increases the efficiency of the process. L. M. Clark.

Evaluation of gas purifying material. H. Asendorf (Gas- u. Wasserfach, 1926, **69**, 957—958).—The iron content of a gas purifying material is not a measure of its adsorptive power. The latter can only be usefully determined by large-scale tests. The results of such a test on a sample of "Lautamasse" from the Vereinigten Aluminium-Werke A.-G. are given. 8½ tons of Lautamasse were used in the purification of 340,000 cub. m. of gas over a period of 4½ months. It then contained 41·70% of free sulphur and 11·27% of Prussian blue. A. B. Manning.

Adsorptive properties of an active charcoal. C. Mazzetti (Annali Chim. Appl., 1926, **16**, 430—438).—A comparison of the efficiency of adsorption of benzene vapour and of various dyes by "Roma" charcoal and other preparations. E. W. Wignall.

Direct determination of nitrogen in illuminating and heating gases. W. Steuer (Chem.-Ztg., 1926, **50**, 860).—The apparatus used consists of two Hempel burettes, one of which contains the gas and the other a slight excess of oxygen over that required for combustion. These are connected by a quartz capillary containing palladium (cf. B., 1925, 802), which is heated first gently and finally more strongly until the volume of the mixed gases becomes constant. The carbon dioxide and oxygen in the burnt gas are absorbed by alkaline pyrogallol, any carbon monoxide liberated at this stage being removed in the usual way, and the residual nitrogen measured. F. R. Ennos.

Influence of the aldehyde content of industrial alcohol on its use for lighting purposes. J. Dehnicke and W. Kilp (Z. Spiritusind., 1926, **49**, 280—281).—To investigate the causes of the gummy residues formed when alcohol is used in lamps, various synthetic mixtures of alcohol, benzene, acetaldehyde, and pyridine were examined. Mixtures which contained both aldehyde and pyridine yielded a resinous gummy residue, but mixtures of alcohol and benzene with aldehyde or pyridine alone yielded only a slight non-gummy residue. This was confirmed by investigating the fall in candle-power over a period of 334 hrs. in lamps using different mixtures. Alcohol which has a high aldehyde content, and has been denatured with pyridine, causes a clogging of the wick by the gummy resins, and a high acid content causes a solution of metallic salts with subsequent deposition on the wick and a consequent reduction of the candle-power of the lamp. A. C. Monkhouse.

Combustible liquids of high organic sulphur content as a source of anti-detonators. Y. Altchidjian (Compt. rend., 1926, **183**, 975—978).—Distillation of the bituminous limestones of Raguse yields heavy fractions up to 350°, which may be used satisfactorily in internal-combustion engines without producing detonation. This is due partly to the presence of a high proportion of hydrocarbons which have undergone a certain amount of degradation towards the unsaturated, cyclic state, and partly to the presence of organic sulphur compounds. Since the at. wts. of the elements of the sixth group, and also the anti-detonating powers of their organic compounds, form approximate geometrical progressions, the anti-detonating powers of these sulphur compounds are evaluated. If they are present in the fraction to the extent of 8—10%, an anti-detonating power equivalent to 0·1% of lead tetraethyl is produced. J. Grant.

Analysis of small quantities of lubricating oils. [The Vogel–Ossag viscosimeter.] G. Meyerheim and F. Frank (Z. angew. Chem., 1926, **39**, 1451—1454).—The Vogel–Ossag viscosimeter has been designed to test small quantities of lubricating oils. It is essentially a modification of the Ostwald capillary viscosimeter, completely enclosed in a water-bath, and so arranged that by means of a pneumatic pump provided a whole viscosity curve, η against t, can be determined from one filling of 15 c.c. of oil. The results compare very favourably with those obtained by the older methods, but the apparatus is not satisfactory for testing dark or very viscous oils. A crucible is also described by the use of which reliable results for flash-points and ignition points, within the limits $\pm5°$, are obtainable with only 10 c.c. of oil. E. Holmes.

Acid value of the "blown" oil and the "tar-forming" value. H. von der Heyden and K. Typke (Petroleum, 1926, **22**, 1288—1289).—The varying proportion of free to combined acid, and the varying molecular weights of the acids formed in different oils account for the absence of a simple relationship between the acid value of the blown oil and the tar-forming value of the oil. In investigating an oil, therefore, both values should be determined. A. B. Manning.

Drying with hot gases. Graulich.—See I.

Coal tars from steamed vertical retorts. Parker.—See III.

Tars and oils from coal. Sinnatt and others.—See III.

Vertical-retort tars. Macleod and others.—See III.

Influence of carbonising conditions on the free carbon content of tar. Hollings.—See III.

Patents.

Heat treatment or carbonisation of coal. Internat. Combustion Engineering Corporation, Assees. of W. Runge and E. A. Packard (E.P. 253,498, 17.5.26. Conv., 12.6.25. Addn. to E.P. 242,621).—Finely-ground coal is preheated to 350° in gravitating down a tower up which hot gases, preferably containing oxygen, are blown. The treated coal does not differ materially in chemical composition from the original coal; the particles, however, have lost all the angular sharpness of the original coal. The preheated coal is then carbonised at 550° by a similar process. The final product is a powdered coke, the particles of which are porous, spheroidal shells of low sp. gr., and contain 8—12% of volatile hydrocarbons. S. Pexton.

Manufacture of vegetable carbon. L. H. and A. H. Bonnard (E.P. 260,666, 4.8.25).—A carbonaceous material is carbonised with magnesium carbonate as activating medium. The magnesium oxide is extracted from the residue by carbon dioxide under pressure, and in the presence of sufficient water to dissolve the magnesium bicarbonate formed. This is recovered from the solution as magnesium carbonate by causing it to react with magnesium oxide in a fresh portion of retorted residue or by heating it. R. A. A. Taylor.

Production of carburetted gas. Frankfurter Gas-Ges., F. P. Tillmetz, and E. Schumacher (E.P. 260,501, 28.4.26).—Water-gas is carburetted by the injection of a mixture of oil or tar vapour and steam, which may be superheated, into the producer at a level where the temperature lies between 700° and 900°. The same process may be used for the enrichment of coal gas. A. B. Manning.

Production of carburetted water-gas. Frankfurter Gas-Ges., F. P. Tillmetz, and E. Schumacher

(E.P. 260,511, 29.5.26. Addn. to 260,501; see preceding abstract).—A gas producer comprising two parts, the upper one being of smaller diam. than the lower, is fed with coke. Each part is provided with an outlet valve through which the "blow" products pass away. The two valves are interconnected so as to fix the relative amounts of blow gases traversing the two sections of the plant, thus enabling the upper part of the charge of coke to reach a temperature of 800° when the lower part has reached 1000°. At this stage steam for making water-gas is introduced into the bottom of the generator, and simultaneously oil vaporised in a current of steam is introduced into the generator between the two sections. The oil is gasified at 800° in a stream of steam and water-gas in the upper part of the producer. S. Pexton.

Manufacture of combustible gas. W. J. Murdock, E. E. Lungrew, and O. B. Evans, Assrs. to Pier Process Corp. (U.S.P. 1,602,242, 5.10.26. Appl., 23.9.24).—A gas producer for gasifying coal and producing relatively rich gas is provided with a central refractory wall. All parts of the annulus of fuel being within radiating distance of the refractory walls, no impermeable core of fuel is formed. The producer is alternately blown and steamed in a vertical direction over substantially the whole of the grate area. S. Pexton.

Process and apparatus for coking coal. Urbana Coke Corp., Assees. of S. W. Parr and T. E. Layng (E.P. 249,886, 27.3.26. Conv., 27.3.25).—Crushed coal is heated in a state of agitation to within a few degrees of the temperature at which it becomes plastic. During preheating the cellulosic constituents, which are devoid of coking properties, decompose with the evolution of water and carbon dioxide. The hot product is transferred in bulk into retorts preheated to 750°. To produce low-temperature coke of high volatile content no additional heating of the retort setting is required. For the production of metallurgical coke it is necessary further to increase the temperature of the retorts. By this two-stage process non-coking coals having a high cellulosic content yield excellent coke. S. Pexton.

Apparatus for carbonising wood. H. Hennebutte and E. Goutal (E.P. 248,376, 24.2.26. Conv., 28.2.25).—Wood is carbonised in a perforated box enclosed within a cylindrical trolley having fluid-tight doors for charging and discharging. The bottom of the perforated box forms a duct with the bottom of the trolley whereby the gases evolved during distillation can pass to the outlet at one end of the trolley. The trolley passes through a tunnel which is heated by furnace gases which enter and leave by ports situated on the opposite sides of the tunnel. The hot gases pass over the trolley, the underside being protected in order to avoid degradation of the products of distillation.
S. Pexton.

Distilling crude oils from shale. C. A. Spotz (U.S.P. 1,601,777, 5.10.26. Appl. 8.6.25).—Shale is delivered by a plunger to the underside of a reticulated tray, which is totally immersed in an enclosed tank of molten tin, or like metal, maintained at 425°. The tray slopes slightly upwards to the exit end of the tank, and the reciprocating motion passes the shale in that direction. As the shale loses gas it disintegrates, passes through the reticulations in the tray to the surface of the metal, and moves towards the exit end of the tank under the action of rotating blades. Means are provided for admitting fresh material and withdrawing spent residues without admission of air. T. S. Wheeler.

Effecting reaction between liquids tending to form tight emulsions. Aktiebolaget Separator, Assees. of F. H. McBerty (E.P. 238,541, 7.8.25. Conv., 16.8.24).—In the purification of lubricating oils from dispersed carbonaceous material, the oil is agitated with an immiscible counter-colloid which causes agglomeration of the carbon particles. The carbonaceous material is partially agglomerated by agitation of the oil with the reagent for a short time, and the oil and the reagent separate easily. By repeating the process the oil is finally purified. S. Pexton.

Dehydrating petroleum emulsions. H. C. Eddy and G. B. Hanson, Assrs. to Petroleum Rectifying Co. of California (U.S.P. 1,602,190, 5.10.26. Appl., 4.1.26). —In pumping an oil well a gas containing a demulsifying agent is introduced into the oil in the well. H. Moore.

Filtration of fluids [lubricating oil]. C. S. Garland and T. E. Beacham (E.P. 260,699, 18.8.25).—Oil is filtered in a pack or edge filter which is maintained at such temperature that any water will pass through with the oil as vapour, and the viscosity of the oil will be greatly reduced. The filtration may preferably be effected by vacuum, so that the water is removed from the oil as vapour, but an occasional back-flow of air under pressure is provided for cleaning the filter.
B. M. Venables.

Removal of sulphur compounds from petroleum oils. L. L. Odom, Assr. to M.O.R. Products Co. (U.S.P. 1,604,235, 26.10.26. Appl., 13.6.24).—The vapours from a still are passed through a column tightly packed with copper fibres of an even and hair-like consistency. Removal of 99·75% of the sulphur content is claimed. W. N. Hoyte.

Refining of petroleum oils. P. McMichael, Assr. to Hydrocarbon Refining Process Co., Inc. (U.S.P. 1,603,701, 19.10.26. Appl., 11.1.24).—Distillates volatile below 225° are agitated first with a solution of alkali, then with 1·5 to 15% of sulphuric acid of strength 70—83%; finally the oil is distilled with steam in the presence of alkali. W. N. Hoyte.

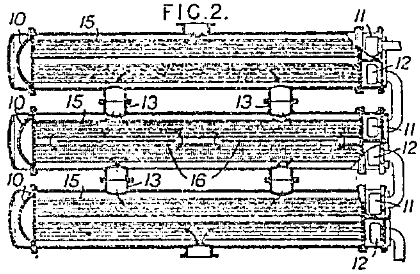
Refining petroleum. W. A. Schmidt, Assr. to International Precipitation Co. (U.S.P. 1,604,424, 26.10.26. Appl., 19.7.20; cf. U.S.P. 1,204,906—7; B., 1917, 38).—The oil to be treated is sprayed into a current of hot gas, and the mixture is led through an electrical precipitator to remove tar. It is then cooled to any desired extent by introduction of cold gas, and is subjected to electrical precipitation. The process of adding gas and precipitating is repeated as required. The permanent gas finally obtained is employed partially to vaporise fresh oil, and partially for cooling purposes.
T. S. Wheeler.

Purification of mineral oil distillates and of paraffin wax. Burmah Oil Co., and R. R. le G. Worsley (E.P. 260,455, 27.1.26).—Any fraction of a petroleum or shale oil, or paraffin wax etc., is refined,

by the action of fuller's earth or other hydrosilicate, while in vapour and at a reduced pressure. The temperature is not more than sufficient to maintain the oil in vapour, and the vacuum as high as practicable, particularly in treating the higher-boiling fractions. The vapours may be purified by contact with increasingly active adsorbent in successive chambers. The oil may be preliminarily washed with sulphuric acid, or with alkali, to remove phenols, and these processes may alternate with the treatment with adsorbent. Inert vapours may be mixed with the oil vapours to reduce the time of operation. H. MOORE.

Apparatus for cooling oil vapours. GRISCOM-RUSSELL Co., Assees. of J. PRICE (E.P. 240,415, 4.8.25. Conv., 29.9.24).—The oil vapour flows zig-zag downwards

FIG. 2.



as shown by the arrows in the figure, and a cooling medium flows through the banks of tubes in succession. The condensate formed in each unit may, if desired, be collected separately. B. M. VENABLES.

Treatment of acid sludge. R. A. HALLORAN, W. N. DAVIS, and G. A. DAVIDSON, Assrs. to STANDARD OIL Co. (U.S.P. 1,604,641, 26.10.26. Appl., 7.8.22).—The sludge, together with steam and water, is continuously passed into a vessel where the mixture is maintained at such a temperature and pressure that hydrolysis takes place. Acid and tar are drawn off from the vessel at required rates. W. N. HOYTE.

Manufacture of stable iron carbonyl compositions. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 206,639, 2.5.25).—The decomposition of iron carbonyl by light can be retarded or avoided by the presence of about 0·1% of a colouring matter (*e.g.*, azo dyes), organic acid, ester, or other organic oxy-compound of high mol. wt. which is capable of acting as a protective colloid, or a solid or liquid hydrocarbon with a b.p. above 275°. In the latter case the hydrocarbon or its mixture used as a solvent should have a much lower b.p. H. ROYAL-DAWSON.

Manufacture of iron carbonyl compositions. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 260,640, 2.5.25).—The composition consists of at least 20% by volume of iron carbonyl in a mixture with liquid hydrocarbon diluent, *e.g.*, benzol or gasoline. H. ROYAL-DAWSON.

Supplementary charges for internal-combustion engines. H. A. KELTY (E.P. 260,676, 5.8.25).—The supplementary charge consists of the vapour of a mixture of alcohol and water which is fed to the carburettor from a container which can be heated by the exhaust gases from the engine. Feed, composition of mixture, etc. are controlled thermostatically. R. A. A. TAYLOR.

Production of hydrogen practically free from carbon monoxide from water-gas (E.P. 259,643).—See VII.

Manufacture of a manure [from peat etc.] (E.P. 260,882).—See XVI.

III.—TAR AND TAR PRODUCTS.

Coal tars from steamed vertical retorts. A. PARKER (J.S.C.I., 1926, **45**, 408—411 T).—In the carbonisation of coal in continuous vertical retorts at high temperatures, as compared with horizontal-retort practice, the coal is submitted to a gradual rise in temperature, and the primary tar products are removed from the top of the retort without having undergone so much degradation by long exposure at high temperatures. Continuous vertical-retort tars may, therefore, be classed as intermediate, in both quantity and composition, between high-temperature and low-temperature tars. The passage of steam upwards through continuous vertical retorts also reduces the amount of secondary thermal decomposition, by removing the products more rapidly from the retort and by ensuring more uniform heating of the coal. In an investigation of the process of steaming the charges in continuous vertical retorts, it was found that with Bothwell washed nuts the yield of dry tar per ton of coal carbonised rose from 13·2 gals. when no steam was admitted to the retorts to 21·2 gals. when the amount of steam was equivalent to 23·7% of the weight of coal. At the same time the amount of "free carbon" in the tar decreased from 1·5 to 0·4%, indicating a reduction in the extent of thermal decomposition. Coal charged in the form of screened nuts more readily allows the flow of gases and steam than coal charged in a more finely-divided condition. In experiments in which different grades of sizes of a particular coal were carbonised under identical conditions, the yield of tar progressively decreased as the sizes of the particles of coal were diminished, and the amounts of naphthalene and "free carbon" in the tars increased, demonstrating that the tar vapours from the smaller grades of coal had undergone greater thermal decomposition. In an examination of the published results of distillation tests of tar, it is almost impossible to make satisfactory comparisons. The reasons are discussed, and a strong plea is put forward for a standard simple laboratory distillation test under specified conditions.

Vertical-retort tar for road purposes. M. BARASH (J.S.C.I., 1926, **45**, 441—448 T).—The use of tars for road purposes is reviewed, and stress is laid on the anti-skid properties of tarred roads as compared with other road surfaces. To the tar producer, vertical-retort tar shows a considerably greater financial return than horizontal-retort tar. A number of gas undertakings are turning out from their own stills residue tars of vertical-retort origin which satisfy the Ministry of Transport (Road Department) Specifications. The essential characteristics which distinguish present-day vertical-retort tars from tars produced in intermittent systems of carbonisa-

tion are given. To the tar refiner the preparation of road tars from vertical-retort tars presents no difficulty. In the majority of cases straight distillation gives residues of the required properties. In some cases, however, as in the case of a tar containing an excess of phenols or tar acids, " topping " has to be carried out to a higher degree than would otherwise be necessary; the residue may then have to be " cut back " to the correct consistency with a decarbolated creosote. The use of road tar prepared from vertical-retort tar has been rapidly extending during the last 10 to 15 years, and it already forms an appreciable proportion of the tars used for road construction and maintenance.

Vertical-retort tar. J. Macleod, C. Chapman, and T. A. Wilson (J.S.C.I., 1926, **45**, 401—1406 t).—The essential differences between tar produced at Glasgow Corporation's Provan Station from Woodall–Duckham continuous vertical retorts, using Lanarkshire coal of moderate coking power, and tar produced by other systems of carbonisation, are indicated. It is shown that, while the yield of motor spirit is lower, the total yield of rectified naphtha is more comparable with that from horizontal-retort tar, though the higher paraffin content of the vertical-tar products renders them useless for the preparation of dyestuffs. The yield of tar acids, tar bases, and creosote is considerably higher than from other carbonising processes, pitch production is lower, naphthalene is found in very small amount, and anthracene in traces only. A comparison of the value of products from one ton of tar, at market prices quoted in April, 1926, from Provan vertical retorts with those from Provan horizontal retorts prior to the installation of the Woodall–Duckham retorts shows the total values to be 131/10·7 and 120/3·7, or on the basis of products from one ton of coal carbonised 9/5 and 7/0 respectively. These estimates take no note of cost of production, but it is claimed that, owing to the absence of salts, less capital outlay for plant is necessary and the saving of steam for heating purposes is considerable. Trouble has been experienced in refining the tar acids, so as to yield high-boiling products (of which the amount present is largely in excess of that from horizontal tars) which do not rapidly darken on keeping, and which, when emulsified with a soap solution, will yield disinfectants capable of maintaining their white colour for an appreciable period. These vertical-tar acids invariably yield a red emulsion which is objectionable for certain specific applications. Investigations with a view to the removal of the substances causing this red colour are described, as is also the method finally adopted involving air-blowing of a hot " strong soda " cresylate which has been found effective in practice. This process also renders the freshly-distilled acid more stable, and the darkening, which is extremely rapid in untreated acid, is very considerably retarded. The material of which the acid still is composed has some bearing on this darkening process; of the less expensive and easily-worked metals or alloys, copper is very satisfactory, and arrangements have been made to use copper-lined mild steel stills in the works. The high yield of creosote compensates for the low pitch, and, owing to the absence of naphthalene and anthracene and its high tar acid content, forms a good timber-creosoting oil. It can be readily washed for the removal of tar acids, yielding a spent oil which is clear and perfectly fluid at ordinary temperatures, which are essential requisites in fuel oil. The prejudice formerly existing against using vertical-retort pitch for briquette making is discussed, results of examinations and comparative analyses with pitches from other sources being given. It is pointed out that the total yield of pitch from the distillation annually of 27,500 tons of vertical-retort tar is regularly disposed of at current market rates. The conclusion arrived at is that, everything considered, vertical-retort tar has many advantages over other tars from the distillers' point of view, refining being easier and cheaper (except in the case of tar acids), and the total value of the derivatives being considerably higher.

Distilled tar for roads. W. G. Adam [with F. W. Robinson] (J.S.C.I., 1926, **45**, 412—415 t).—The employment of distilled or refined coal tar for road-making continues to increase in spite of criticisms. The chief faults of tar in the past have been mainly due to the use of improperly refined tar, frequently of unsuitable consistency for the work in hand, and in part due to careless laying. A specification is given based upon experience, and designed to cover only such products as will give satisfactory results in practice when applied under correct conditions. The specification is more stringent than the existing Road Board Specifications, particularly in the clauses relating to the naphthalene and tar acids. It will be possible for the tar distilling industry to supply all the tar required to this specification. Information on the toxic nature of washings from tarred roads has been considerably extended since the days of the Alresford experiments, in which a material was laid down which just conformed (except for the naphthalene content, which fell widely outside the limit) to the Road Board Specification. It has now been found that the toxicity of a given tar decreases with increasing viscosity, and that soft pitches in general are non-toxic under the experimental conditions. The toxic effects of tars become far less evident as the proportion of lower boiling point constituents is reduced. Several processes are now available for preparing tars, which it is claimed are non-toxic to fish life. The real damage caused by drainage from tarred roads has been much exaggerated, and trade effluents are now being recognised to be far more important factors. The best conditions for the application of tars by spraying are considered. With reference to emulsions, it is suggested that the advantages of the ease of application are not sufficient to compensate for the extra cost of the material and the lack of permanency of the results. Figures are given to illustrate the importance of grading the aggregate for road-making to obtain a solid resilient road, free from voids. It is considered that the tar distiller should further assist the surveyor by price stabilisation, and by increased delivery service. Under the latter heading it is proposed that barrels should be replaced by road tanks conveying the hot tar direct from the distillery to the road, these delivery tanks being equipped with spraying and brushing gear.

Tars and oils produced from coal. II. F. S. Sinnatt, J. G. King, and W. H. Linnell (J.S.C.I., 1926, **45**, 385—393 t; cf. *ibid.*, 1925, 413 t).—A series of

investigations on the tar obtained by the carbonisation of a medium caking British coal at temperatures between 400° and 700° are described. Medium-scale steel retorts were used having a maximum capacity of 200 lb. of coal. The yield of tar was found to increase gradually with temperature to a maximum at 550°, the yield at this point amounting to 17·65 gals. per ton of coal. The sp. gr. of the tar also increased gradually up to 550°; above this temperature the rate of increase became greater. A study of the gas yields and of the nature of the tars produced indicated that a critical carbonising temperature occurred at about 550°, *e.g.*, the lighter fractions from the tar (up to 230°) and the middle fractions increased to a maximum at 500°, a marked acceleration in pitch formation occurred above 500°, and the phenols and unsaturateds showed an accelerated increase above 550°. The percentage of saturated hydrocarbons in the tars decreased steadily from 29·8% of the 400° tar oil to 13·4% of the 600° tar oil and to 6·2% of the 700° tar oil. Low-temperature tar obtained in the "commercial" scale distillation of Dalton main coal contained phenols 26·15, phenol 1·17, bases 2·74, neutral oils 41·6, saturated hydrocarbons 8·24, solid paraffins 0·12%. These results are in general agreement with those obtained in previous investigations (Parrish & Rowe, J.S.C.I., 1926, **45**, 99 T; Brittain, Rowe, and Sinnatt, *ibid.*, 1925, **44**, 412 T). Small-scale work on the hydrogenation of coal has confirmed previous results. A liquefaction of 50—60% is possible with the average British bituminous coal. The light oils obtained by this process contain appreciable amounts of benzene, toluene, and xylene, and in this respect differ markedly from those produced by low-temperature carbonisation.

Influence of carbonising conditions on the free carbon content of tar. H. Hollings (J.S.C.I., 1926, **45**, 406—408 T).—The free carbon content of tar from the carbonisation of Durham coal in horizontal retorts is correlated with the weight of charge, the temperature of the combustion chamber surrounding the retorts, and the cross-sectional area of the retort. The weight of charge per cub. ft. of retort space determines the amount of free space within the retort. With temperatures between 1320° and 1376°, the free carbon content increased from 12·1 to 20·8% as the weight of charge was decreased from 35·5 to 29·0 lb. per cub. ft. In a slightly deeper retort a reduction in the weight of charge from 31·0 to 28·4 lb. resulted in an increase in the free carbon content from 20·8 to 24·6% notwithstanding a fall in temperature from 1348° to 1334°. When the weight of charge was maintained at about 29·0 lb. per cub. ft. a reduction in temperature from about 1330° to 1253° resulted in a decrease in the free carbon content from about 22 to 12·3%. With any given retort the most important factor affecting the quality of the tar is the weight of each charge of coal. Any attempt to improve an unsatisfactory tar by reducing the carbonising temperature necessitates a reduction of the temperature below that which is required for the maintenance of such throughputs of coal as are essential for economical working. An increase in the weight of charge results generally in improved working conditions. In an experimental setting retorts 24 in. by 18 in. were set alongside retorts 21 in. by 15 in. in cross-section. Each retort was fully charged with the same coal, and was heated to the same external temperature. The tar from the deeper retorts contained 18·8% of free carbon, that from the smaller retorts contained 9·8%. It was necessary to carbonise the deeper mass of coal in the deeper retort for two hours longer than was necessary for complete carbonisation in the smaller retort. During this extra time the volatile products from the centre of the large coal charge were subjected to high-temperature treatment, while the gas velocity in the retort was relatively low. This resulted in the deposition of solid decomposition residues upon the coke and in the formation of free carbon in the free space.

Consistency of "bitumen mixtures." H. M. Spiers (J.S.C.I., 1926, **45**, 399—401 T).—When consistency is measured by means of Hutchinson's tar tester, the relation between the consistency of bitumen mixtures and their bitumen content may be expressed by means of the formula $dc/d\,B/T=k\,C$, which on integration yields the relationship $\log C_1 - \log C_2 = K\,(B_1/T_1 - B_2/T_2)$, in which C_1, B_1, T_1 are the consistency, bitumen content, and tar content of one mixture and C_2, B_2, T_2 are the corresponding values for another mixture made from the same tar and bitumen. K is a constant the value of which varies at least within the range 1·4 to 3·2, and depends on the nature of the tar, on the extent to which it has been topped, and on the nature of the bitumen (or pitch) added. A nomogram based on the above relationship is reproduced in the original, and facilitates the control of the consistency of mixtures prepared industrially.

Variation of Hutchinson consistency of tars with temperature. H. M. Spiers (J.S.C.I., 1926, **45**, 396—399 T).—Consistency varies with temperature according to the relationship $dc/dT = -kC$, which on integration yields the formula $\log C_2 - \log C_1 = K\,(T_1 - T_2)$. In the equation, C_1 and C_2 are the consistency values at two known temperatures T_1 and T_2, consistency being measured in seconds and temperature in degrees F., K is a constant depending chiefly on the naphthalene content of the tar, and ranging from 0·0334 in the case of tars rich in naphthalene to 0·0502 when the naphthalene content is low. The formula applies also to "bitumen mixtures." A nomogram enables the consistency of a tar at the standard temperature of 77° F. to be ascertained when the consistencies at two known temperatures have been determined, and its use avoids the necessity of cooling the tar to the standard temperature, and enables the consistency at any desired temperature within a definite range to be estimated.

Evaluation of pitch. H. F. Taylor (J.S.C.I., 1926, **45**, 417—424 T).—*Binding power.* The tensile strength of pitch was tested in various ways, but it was found that the pitch invariably broke where the jaws of the testing machine gripped it, and the results are considered unreliable. Coal-tar pitches averaged about 80 lb. per sq. in. In the test considered most likely to give concordant results comparable with works practice, briquettes are prepared by heating 105 g. of the sand standardised for cement testing by the British Engineering Standards Association with 5% more pitch than will fill the interspace, and casting the mixture in a B.E.S.A. mould. The tensile strength of the briquette

is determined in a cement testing machine. Six pitches were tested under varying conditions. Vertical-retort pitch briquettes averaged 250—300, horizontal 350—450, low-temperature 200 lb. per sq. in. *Range of plasticity and elasticity.* A small mechanical apparatus is described, in which the principle of the "twist-point" test is used. The torsion is applied by a weight suspended from a thread running over a pulley and round a bobbin attached to a holder which grips one end of the block of pitch ($2 \times \frac{1}{2} \times \frac{1}{2}$ in.). A dial graduated in degrees of arc indicates the amount of the twist. A ball and ring "fusing" point apparatus is attached, and the difference between the "fusing" temperature and the temperature at which the pitch starts to twist gives an indication of the range of plasticity. Four types of coal-tar pitch gave ranges of about 40° C., soft and hard hardwood pitch 29° and 49°, and petroleum bitumen 56°. If the weight responsible for the torsion is suddenly lifted a recoil occurs, and an elasticity test is based on this. The recoils which occurred when 500 g. had twisted the pitch 360° of arc at the twist point temperature were: horizontal- and vertical-retort pitches, 10° arc, low-temperature pitch 18°, coke-oven pitch 21°, blast-furnace pitch 30°, hard hardwood pitch 88°, petroleum bitumens 120° and 130°. The effect of the length of time which had elapsed since casting the mould and of twisting the pitch a number of times was investigated. The elasticity of low-temperature pitch can be considerably increased by treatment of tar or pitch with sulphur.

Plea for standardisation of analytical methods in coal tar products specifications. W. G. ADAM (J.S.C.I., 1926, **45**, 393—396 T).—Although the desirability of rigidly standardising the methods of analysis of metallic and inorganic products may be debatable, its necessity can hardly be denied in the case of organic commercial products the valuation of which is almost invariably carried out by the observation of physical properties rather than by true chemical analysis. The four chief methods employed in the examination of coal tar products are the observation of (*a*) boiling points or distillation ranges; (*b*) crystallisation or setting points; (*c*) melting or softening points; (*d*) specific gravity. In some few cases fractionation is used, whilst in others, *e.g.*, the determination of *m*-cresol and anthracene, true analytical methods are employed. The use of retorts for the determination of boiling points and distillation ranges is open to very grave criticism; it is suggested that the vessel used should be a standard distilling flask, the standard Engler flask used in the petroleum industry, and adopted to some extent by the B.E.S.A. Further matters requiring attention are the methods of heating and the thermometers employed. For creosote distillations the American authorities have recognised the shortcomings of the retort, and have adopted a retort flask, which has removed many of the disadvantages of the older method. The defects of many of the older methods of testing pitch have been demonstrated, and it is possible that the Kraemer and Sarnow method for determining softening point and the Brookman-Mück test for volatile matter will be adopted. It is desirable to standardise existing methods as far as possible rather than to introduce new ones.

Coal-tar disinfectants. TAIT.—See XXIII.

Acid value of the "blown" oil and the "tar-forming" value. VON DER HEYDEN and TYPKE.—See II.

PATENTS.

Manufacture of bituminous product. D. B. W. ALEXANDER (U.S.P. 1,603,502, 19.10.26. Appl., 4.5.26). —A slurry of finely-divided mineral matter and kerosene is added to a charge of petroleum, and distilled.

B. W. CLARKE.

Removal of tar acids from ammonia liquor. H. W. ROBINSON and D. W. PARKES (E.P. 260,686, 7.8.25, 9.10.25, and 29.3.26).—The liquor is neutralised by a mineral acid. Carbon dioxide (in vitiated airs etc.) may serve for this purpose. The liberated tar acids are then absorbed by active carbon, which is separated from the liquor and treated for the recovery of the tar acids.

R. A. A. TAYLOR.

Treatment of acid sludge (U.S.P. 1,604,641).—See II.

Production of carburetted gas (E.P. 260,501).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Colours in foodstuffs. DRAKE-LAW.—See XIX.

PATENTS.

Manufacture of derivatives of naphthaquinones. SOC. ANON. DES MAT. COL. ET PROD. CHIM. DE ST.-DENIS, R. LANTZ, and A. WAHL (E.P. 246,482, 21.1.26. Conv., 21.1.25).—1-Imino-4-arylimino-2-hydroxynaphthaquinones are prepared by the simultaneous oxidation by air, pure oxygen, or other oxidising agents of 1-amino-2-naphthol or its 4-sulphonic acid and arylamines, or by condensing nitroso-β-naphthol with arylamines. *E.g.*, 52 pts. of 1-amino-2-naphthol-4-sulphonic acid, 14 pts. of anhydrous sodium carbonate, and 25 pts. of aniline are dissolved in 800 pts. of water. The cold solution is stirred and sodium hypochlorite gradually added until there is excess of chlorine and the precipitate no longer increases. The product is purified by dissolving it in cold dilute sodium carbonate, and reprecipitating the filtered solution at once by bicarbonate. The orange 1-*imino*-4-*phenylimino*-2-*hydroxynaphthaquinone* so obtained is identical with the product formed by dissolving 30 pts. of nitroso-β-naphthol in 300 pts. of aniline and 30 pts. of benzene, boiling for 12 min., and cooling. The former method may also be used for the preparation of the 2-hydroxy-1 : 4-naphthaquinonediarylimines described in E.P. 206,142 (B., 1924, 902) from 1-arylamino-2-naphthol-4-sulphonic acids. Thus 1-phenylamino-2-naphthol-4-sulphonic acid (E.P. 206,150; B., 1925, 200) when oxidised together with aniline in slightly alkaline solution by agitation with air yields 2-hydroxy-1 : 4-naphthaquinonediphenylimine. The action of acids on the above products replaces the group in position 1 by oxygen, thus forming 2-hydroxy-1 : 4-naphthaquinone-4-arylimines identical with those described by Boeniger (Ber., 1894, **27**, 23). Again, by the action of arylamines, the group in position 1 is replaced by the arylimino-group with formation of 2-hydroxy-1 : 4-naphthaquinonediarylimines.

A. DAVIDSON.

Dyes and dyeing. [Red flavanthrone derivative.] B. WYLAM, J. E. G. HARRIS, J. THOMAS, and SCOTTISH

Dyes, Ltd. (E.P. 260,638, 30.4.25).—Leucoflavanthrone is treated with an alkylsulphuric halide and a tertiary base at temperatures above about 50°, or the alkylsulphuric halide may be mixed with the tertiary base in the cold, leucoflavanthrone added, and the mixture heated at above 50°. The quantity of tertiary base used may be higher than the molecular equivalent of the alkylsulphuric halide. *E.g.*, 7·5 pts. of methylsulphuryl chloride are stirred, with cooling, into 18 pts. of dry light pyridine under carbon dioxide. 3 pts. of dry leucoflavanthrone are added, and the temperature is raised to 60° during ½ hr., then kept at 60° for ½ hr. more. The yellowish-red melt is poured into 200 pts. of water containing 8 pts. of ammonia solution (*d* 0·880), whereby a reddish solid is precipitated. This product may be used directly for dyeing in faintly alkaline solution, or it may be dissolved in 1½% sodium carbonate solution, aerated to remove unchanged leucoflavanthrone, filtered, and salted out. The product can be dyed or printed on cotton or other textile fibres by hydrolysis and oxidation. Cf. E.P. 247,787 (B., 1926, 403), which describes the preparation of a red flavanthrone derivative by another process. The green flavanthrone derivative of E.P. 245,587 (B., 1926, 235) is excluded from this patent.

A. Davidson.

Dyes and dyeing. [Stable water-soluble vat dye derivatives.] B. Wylam, J. E. G. Harris, J. Thomas, and Scottish Dyes, Ltd. (E.P. 260,647, 4.6.25).—Stable water-soluble derivatives of vat dyes are made by treating the vat dyes with an alkylphosphoric halide or phosphorus oxychloride in presence of a metal, such as zinc dust or copper bronze, and a tertiary base such as pyridine, quinoline, or dimethylaniline. *E.g.*, 30 pts. of methylphosphoryl dichloride are stirred, with cooling, into 150 pts. of pyridine. An intimate mixture of 30 pts. of indanthrone and 30 pts. of zinc dust is added, followed by 60 pts. of methylphosphoryl dichloride. The temperature is allowed to rise to 40°, and, after stirring for 20 min. at this temperature, 1000 pts. of water are added. The mixture is poured into 2000 pts. of water, filtered, and the paste washed with water. The product is a stable, light blue substance soluble in alkalis, and oxidisable apparently to indanthrone by acid oxidising agents. Products obtained in this manner can be dyed or printed on cotton by methods such as those described in E.P. 245,587 and 247,787 (B., 1926, 235, 403). Cf. also E.P. 248,802 (B., 1926, 403). A. Davidson.

Manufacture of chromium compounds of azo-dyestuffs. O. Y. Imray. From Soc. Chem. Ind. in Basle (E.P. 260,830, 22.2.26).—Azo-dyes containing groups capable of being chromed are treated with chromic chromates (cf. Gmelin–Kraut, 7th Edn., III, pt. 1, p. 357, etc.). *E.g.*, 37·8 pts. of the sodium salt of the dye obtained from diazotised 4-chloroaniline-3-sulphonic acid and salicylic acid are dissolved in 1200 pts. of boiling water, and a 7% chromic chromate paste is added, made from sodium dichromate and sodium thiosulphate (*loc. cit.*, p. 359) containing chromium equivalent to 22·8 pts. of Cr_2O_3. The mixture is boiled under reflux for a long time. Chromium oxide is filtered off and the dye isolated by concentration and salting out. The products obtained by this method dye wool in an acid bath very even tints fast to light and fulling.

A. Davidson.

Preparations [for generating diazonium salt solutions] suitable for developing baths for use in dyeing (E.P. 260,682).—See VI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Effect of dry cleaning on silks. M. H. Goldman, C. C. Hubbard, and C. W. Schoffstall (U.S. Bureau Standards Tech. Papers No. 322, 1926, **20**, 605—634).—Tin-weighted and unweighted specimens were treated with dry-cleaning agents containing benzine soap, also with acid and alkaline perspiration solutions followed by dry cleaning. No deterioration results from use of the dry-cleaning agents or from subsequent conditioning for over two months at 21° F. and 65% R.H., even after perspiration treatment. Sunlight causes greater tendering in weighted than in unweighted silks, the extent being increased by previous perspiration treatment. Damage, which is attributed to the production of acid in the fabric, is expressed in terms of the breaking load of the untreated material. B. P. Ridge.

Adsorption of malodorous substances formed during the manufacture of sulphate-cellulose. O. Routala & A. V. Jäättelä (Cellulosechem., 1926, **7**, 169—173).—The development of the sulphate pulp industry has been hindered by the extremely unpleasant odour of the exit gas, due chiefly to the presence of methyl mercaptan. This substance is adsorbed by vegetable substances such as wood, straw, etc., and this appears to be the most promising method for dealing with the nuisance (Schwalbe, B., 1922, 747 a). Wood which has been used for adsorbing methyl mercaptan does not give a higher yield of this substance in the sulphate process, as the mercaptan reacts with atmospheric oxygen in the pores of the wood. In the present research moisture is shown to have an important effect on the adsorption of mercaptan by sawdust from conifers, the best results being obtained with sawdust containing 50% of moisture. Sawdust absorbs 14 times as much as wood chips of the same moisture content, and the removal of all mercaptan from the exit gas of a sulphate pulp factory is possible by the use of the quantity of sawdust normally obtained as a by-product. The power of adsorption can be increased by treating the sawdust with aqueous solutions of various substances such as ammonium persulphate, sodium nitrite, sodium perborate, hydrogen peroxide, sulphate black liquor, ammonia, alkalis, or copper, mercury, or lead salts. Straw and peat, animal and wood charcoals are all better adsorbents than sawdust, the effectiveness of the former two materials being largely influenced by the moisture content. W. J. Powell.

Alkali-cellulose. III. E. Heuser and R. Bartunek Cellulosechem., 1926, **7**, 169; cf. B., 1925, 311).—The authors disagree with Hess (Z. angew. Chem., 1925, **38**, 230) regarding the constitution of the sodium hydroxide-cellulose complex. They have shown that the amount of sodium hydroxide taken up by cellulose from concentrated solutions (35—50%) only slightly exceeds that required for the formula $(C_6H_{10}O_5)_2,NaOH$. Higher results are obtained with very concentrated solutions, but these are due to precipitation of sodium hydroxide by the cellulose. The amount of potassium hydroxide taken up from the most concentrated solutions

is never more than that required for $(C_6H_{10}O_5)_2$,KOH, and there is no reason to expect a difference in the behaviour of the two alkalis. It is therefore doubtful whether a compound of the formula $C_6H_{10}O_5$,NaOH can exist. Contrary to Hess, the authors find that the swelling action of sodium hydroxide, though reduced, is not prevented by the presence of alcohol, and the compound $(C_6H_{10}O_5)_2$,NaOH is still formed, unless the concentration of alcohol is high enough to prevent sufficient dissociation of the sodium hydroxide in solution, but the formation of alkali-cellulose may be masked by the increased adsorption of sodium hydroxide caused by the addition of alcohol. W. J. Powell.

Ripening of viscose. O. Faust, E. Graumann, and E. Fischer (Cellulosechem., 1926, 7, 165—166).—The work of Bernhardt (Kunstseide, 1926, 8, 173) and of Heuser and Schuster (B., 1926, 399) on the method for the determination of the degree of ripening of viscose, based on the reaction between iodine and sodium cellulose xanthate, is discussed, and the following procedure is recommended: 25 g. of viscose are made up to 250 c.c. with distilled water. 25 c.c. of this solution are diluted with 2 litres of water, made just acid with *N*-acetic acid solution (1 drop over the neutral point), and then an excess of 0·1*N*-iodine solution is added. These operations must be carried out quickly in view of the rapid ripening of viscose in dilute solution and the decomposition of the cellulose xanthate by dilute acetic acid. The time required (5—10 sec.) should be the same in every determination. The dixanthate formed on addition of iodine is apparently stable towards acetic acid. After ½ hr. the excess of iodine is titrated with 0·1*N*-sodium thiosulphate solution. The determination is then repeated with sulphuric acid in place of acetic acid, the difference between the two results being a measure of the degree of ripening. The method gives concordant figures, and is suitable for the control of manufacturing operations. An unsuccessful attempt was made to find an acid of lower dissociation constant than acetic acid, which would decompose sodium trithiocarbonate but not cellulose xanthate. The hydrogen-ion concentrations required in the two reactions were of the same order, although there was a difference in the reaction velocities. W. J. Powell.

Industrial resinates [for gumming paper etc.]. Uzac.—See XIII.

Culture of the bacillus of retting of flax. Oméliansky and Kononov.—See XVIII.

Alloys [for jets used in spinning artificial silk]. Dreaper.—See X.

Patents.

Impregnating [fabrics] with rubber (E.P. 260,652).—See XIV.

Manufacturing a rubberised fibre composition (U.S.P. 1,603,318).—See XIV.

Making a rubberised fibre composition (U.S.P. 1,603,335).—See XIV.

Making cellulose esters of carboxylic acids (U.S.P. 1,604,471).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing of vat colours. G. O. Mitchell (J. Soc. Dyers and Col., 1926, 42, 374—376).—For the hot-dyeing of yarn with such colours as Duranthrene Golden Yellow Y and 2RT, Orange 4R, Blue 3GT, GCD, CC, and RD extra, Hydron Yellow NF, Pink FF and FB, Caledon Jade Green, and Indanthrene Grey 3B and Green 2G, the dye-bath conveniently contains 1—20 lb. of colour paste, 10—20 lb. of caustic soda (*d* 1·38), and 1¼—4 lb. of hydrosulphite powder in 150 gal. of water at 50—60° for 50 lb. of yarn. With cold-dyeing colours, such as Indanthrene Corinth RK, Caledon Yellow 3G, and Red FF, Duranthrene Brilliant Violet R, Olive R, Red BN, and Red Violet 2RN, only about half the amount of sodium hydroxide is required. The actual dyeing in this case is carried out at 20—25°, and for medium or heavy shades the addition of Glauber's salt is necessary. The distinction between hot- and cold-dyeing colours is not rigid. Thus Caledon Jade Green is best dyed at 25—30°, and Duranthrene Brilliant Violet R and Olive R, Indanthrene Brown R and G can be used as shading colours by the hot-dyeing process. Similar but more concentrated baths containing less sodium hydroxide are used for the dyeing of piece goods, and concentrated stock solution of the colour is with advantage prepared from 8 lb. of colour paste, 5 lb. of caustic soda (*d* 1·38), 1·75 lb. of hydrosulphite, and 10 gal. of water at 50—55°. Precautions are necessary against over-oxidation, and with mixtures of Duranthrene Blues sodium perborate can be used for oxidising. Lists are given of the colours most suitable for hot- and cold-dyeing of piece goods. In the dyeing of viscose silk with vat colours, skilful and rapid manipulation of the yarn is one of the most important points. R. Brightman.

Problems of modern calico-printing technique. J. R. Hannay (J. Soc. Dyers and Col., 1926, 42, 369—374).—The author regards the vat dyestuffs as strongly individualistic in their behaviour on dyeing, and further information is desirable regarding their individual properties, particularly in regard to the heat generated in the formation of the leuco-compounds from the original dyes, and the effect of temperature on the reverse reaction, for the production of the best results. More effective stripping agents for vat colours are also required, and the extension of the Indigosol range of vat colours demands the development of a special technique for these colours. Reasons are given in support of the assertion that no existing range of printing colours giving a full palette can be properly issued with an unlimited guarantee. Thus the penetration of the fibre by the dyestuff, and in consequence the fastness of the latter to scouring, depends inversely on the fineness of the material. R. Brightman.

Patents.

Preparations [for generating diazonium salt solutions] suitable for developing baths for use in dyeing. J. Y. Johnson. From Badische Anilin u. Soda Fabr. (E.P. 260,682, 5.8.25).—A diazotisable amine or a dry neutral salt of it is mixed in about equal molecular proportions with a nitrite. A water-soluble neutral salt may be added as diluent. Solid compounds of acid nature such as bisulphates, naphthalene-

trisulphonic acid, etc. may also be added. It is advantageous to add to the mixture a wetting agent such as Turkey-red oil or condensation products of formaldehyde with phenol- or naphthalene-sulphonic acids. Such mixtures are stable if protected from moisture, and yield solutions of diazonium salts when dissolved in water.

A. DAVIDSON.

Dyes and dyeing. [Red flavanthrone derivative] (E.P. 260,638).—See IV.

Dyes and dyeing. [Stable water-soluble vat dye derivatives] (E.P. 260,647).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Modern saltpetre industry and the nitrate problem. A. SCHAARSCHMIDT (Z. angew. Chem., 1926, **39**, 1457—1460).—Mainly a re-statement of the results of work already published on the interaction of the oxides of nitrogen with olefines and aromatic hydrocarbons and their derivatives, together with a discussion of possible industrial developments (see A., 1924, i, 380; 1925, i, 123, 532, 877; ii, 227).

E. HOLMES.

Preparation of calcium cyanide. H. H. FRANCK and C. FREITAG (Z. angew. Chem., 1926, **39**, 1430—1432).—Following the method of Bergstrom (A., 1924, i, 949) the preparation of calcium cyanide is carried out in liquid ammonia at the ordinary pressure and at temperatures of −35° to −80°. On adding electrolytic calcium to a solution of hydrogen cyanide in liquid ammonia a vigorous reaction takes place and a blue-grey paste results. The product, purified by washing with liquid ammonia, proves to be *diammonium calcium cyanide*, the yield being quantitative. This compound, which also results from the use of calcium nitrate in the above reaction, is a blue-white, apparently amorphous powder. It is stable *in vacuo* at 100°, begins to lose ammonia at 150—160°, and by heating *in vacuo* for 2 hrs. at 180° yields a free calcium cyanide which appears to be purer and of greater density than that of Metzger (B., 1926, 272).

E. HOLMES.

Germanium. XVII. Fused germanium dioxide and some germanium glasses. L. M. DENNIS and A. W. LAUBENGAYER (J. Physical Chem., 1926, **30**, 1510—1526).—Germanium dioxide, m.p. approx. 1100° (cf. Müller and Blank, A., 1925, ii, 145), was obtained as a fused, clear, colourless, transparent glass by heating at 1450—1500° in a high-frequency induction furnace, with frequent stirring, for 30 min. The product contained traces of silicon, aluminium, iron, and copper up to 0·2%, and had n_C 1·60304, n_D 1·60696, n_F 1·61762, *i.e.*, 10% higher than the values for fused silica (Gifford, Proc. Roy. Soc., 1910, 84, 193). The dispersive power is greater than that of fused silica, but the partial dispersion ratios are approximately the same. The effective refractivity is lower, the transmission is the same, except in the ultra-violet, when it is much lower, the density and thermal expansion are also much higher than obtains with quartz. It is less resistant to water and acids, but can be fused and freed from bubbles at lower temperatures. At high temperatures, carbon, organic compounds, and carbon monoxide rapidly reduce it, and the glaze of a porcelain crucible is attacked. Germanium light flint, crown, barium crown, and boro-crown glasses were prepared by substituting equimolecular amounts of germanium dioxide, prepared by the method of Dennis and Johnson (A., 1923, ii, 570), for silica in the corresponding silicate glasses. The fusions were made in unglazed porcelain crucibles heated in small, regenerative gas furnaces of the Hempel type at 1200—1400° for 3 hrs. Crucibles made of platinum, alundum, quartz, or glazed porcelain were unsuitable. Compared with silicate glasses, those of germanium possess higher refractivity and dispersion, a transmission which is approximately the same in the visible and infra-red portions of the spectrum, but lower in the ultra-violet, and partial dispersion ratios which are nearly equal. The densities and thermal expansions are higher, the hardness is the same, but the softening temperatures are lower. Devitrification occurs more readily. Durability and probably resistance to weathering are somewhat less than is the case with the corresponding silicate glasses. The optical properties of the germanium glasses suggest that they could be advantageously used in the construction of optical systems. Satisfactory glasses, intermediate in properties between the two types, can be made by replacing any part of the silica with germanium dioxide in the ordinary silicate glasses.

L. S. THEOBALD.

Determination of carbon dioxide in carbonates. J. R. I. HEPBURN (Analyst, 1926, **51**, 622—624).—From 0·15—0·3 g. of the carbonate is weighed into a test tube, covered with distilled water, and placed in a 750 c.c. filtration flask containing 50 c.c. of 0·1*N*-baryta solution. A separating funnel containing 3*N*-hydrochloric acid is fixed on the flask, which is evacuated to a pressure of 2 cm. of mercury, the clip to the air pump is then closed, and the acid cautiously run into the flask. The evolution of carbon dioxide, which is absorbed by the baryta solution, is assisted by the partial vacuum, and ordinary temperatures usually suffice. After remaining for 12—24 hrs. the remainder of the baryta is titrated with 0·1*N*-oxalic acid. Results were found to be within 0·5% of the theoretical value.

D. G. HEWER.

Removal of oxygen from commercial carbon dioxide. N. H. HARTSHORNE and J. F. SPENCER (J.S.C.I., 1926, **45**, 474 T).—The increase in the life of chromous chloride when used as an oxygen absorbent, effected by the addition of amalgamated zinc, is discussed.

Effect of acids on the mechanical strength of timber. ALLIOTT.—See I.

Analysis of lime. BLUNT.—See IX.

PATENTS.

Method of making arsenic acid. H. W. AMBRUSTER (U.S.P. 1,603,308, 19.10.26. Appl., 9.4.25).—Arsenious oxide and nitric acid are gradually fed into a chamber and thoroughly agitated, the mixture meanwhile being slowly removed to another chamber.

H. ROYAL-DAWSON.

Mixtures of alkali hypochlorite and alkali chloride in solid form. A. OPPÉ (E.P. 249,510, 12.3.26. Conv., 23.3.25).—A highly concentrated and atomised

solution of caustic soda is sprayed by means of gaseous chlorine into an empty chamber, or by blowing powdered caustic soda dust into a chamber containing chlorine, the products being ultimately dried by passing a current of air into the reaction chamber.

H. ROYAL-DAWSON.

Manufacture of chrome alums. W. CARPMAEL. From I.-G. FARBENIND. A.-G. (E.P. 260,885, 28.5.26).—The crystallisation of chrome alum is accelerated if a solution of chromium sulphate, acidified with sulphuric acid, is heated at 30—45°, the requisite amount of potassium sulphate is then added, and the solution cooled rapidly, with agitation. W. G. CAREY.

Recovering vanadium compounds from iron ores containing vanadium and titanium. B. PER F. KJELLBERG (E.P. 260,661, 30.7.25).—The finely-ground ore, or concentrate, is mixed with a halogen compound of an alkali metal or of calcium or magnesium, formed into briquettes, and roasted for some hours at 950—1050°, at which temperature the vanadium compounds are not fully oxidised to the V_2O_5 stage, thus avoiding the formation of sparingly soluble compounds. The roasted mass is treated with dilute hydrochloric acid, which extracts 90% of the vanadium compounds. The vanadium from the solution is precipitated by the addition of lime. M. E. NOTTAGE.

Treating [recovering] vanadium compounds. H. BLUMENBERG, JUN., Assr. to STOCKHOLDERS SYNDICATE (U.S.P. 1,604,630, 26.10.26. Appl., 25.11.25).—Crude phosphate rock containing up to 0·5% V_2O_5 is treated with ammonium nitrate and sulphur dioxide, and the solution obtained is freed from all base metals except alkali metals, and agitated for 6—24 hrs. with oxy-cellulose, when a vanadium compound of the carbohydrate separates. The vanadium is recovered by ignition. T. S. WHEELER.

Simultaneous recovery of nitric oxide, and the production of alkali nitrate and hydrochloric acid. W. H. ROSS and A. L. MEHRING (U.S.P. 1,604,660, 26.10.26. Appl., 7.10.25).—A stream of nitrogen peroxide mixed with air is brought into contact with a saturated solution of an alkali chloride.

H. ROYAL-DAWSON.

Production of hydrogen practically free from carbon monoxide from water-gas. G. CICALI (E.P. 259,643, 15.6.25).—Water-gas is freed from carbon dioxide and hydrogen sulphide, and then further purified to remove the bulk of the carbon monoxide. A volume of free nitrogen equal to the volume of carbon monoxide removed is added to the purified gas, and the mixture is compressed, cooled, and passed into a liquefying machine provided with a fractionating column. The most readily liquefiable component, viz., the carbon monoxide, liquefies in the greatest proportion. The hydrogen and nitrogen contain smaller and smaller quantities of carbon monoxide in the upper parts of the rectifying column. Superimposed upon the rectifying column are a series of tubes surrounded by a mixture of liquid carbon monoxide and nitrogen, supercooled by evaporation at approximately atmospheric pressure. The hydrogen and nitrogen containing traces of carbon monoxide pass through these tubes, in which the bulk of the nitrogen and the last traces of carbon monoxide condense. The hydrogen, free from carbon monoxide but containing some nitrogen, leaves the refrigerating machine by a coiled pipe surrounding the tube by which the gas to be purified enters. S. PEXTON.

Continuous crystallising apparatus (E.P. 260,691). **Drying devices for granular material and the like** (E.P. 260,896).—See I.

Iron carbonyl (E.P. 206,639 and 206,640).—See II.

VIII.—GLASS; CERAMICS.

Determination of soluble alkali in glass. E. FISCHER and W. TEPOHE (Glastech. Ber., 1926, 4, 137—142; Chem. Zentr., 1926, II, 2010).—A modification of Mylius' method in which methyl-red is used in place of iodoeosin.

Constitution of porcelain. R. SCHWARZ and H. MERCK (Z. anorg. Chem., 1926, **156**, 1—16).—The literature relating to the nature of the crystalline aluminosilicate formed on heating clays to high temperatures is reviewed (cf. also Hyslop, Trans. Ceram. Soc., 1925, 24, 402). Analytical data are presented in support of the assumption that mullite, rather than sillimanite, is the silicate formed in fired porcelain. The method used to isolate mullite closely resembled that previously applied by Bowen and Greig (B., 1924, 512). Analyses of unglazed porcelains, fired from one to six times in a porcelain oven, showed that the percentage of mullite increased from an average of 3·7 to 13·0%. Similarly, in Zettlitz kaolin, fired for 2 hrs. at 1400° and for 4 hrs. at 1500°, the amount of mullite increased from 30·2% to 44·7%. The resistance to the action of chemicals was not appreciably affected by repeatedly firing porcelain. Porcelain fired four times was somewhat more resistant to abrupt changes of temperature than that once fired. Six-fire porcelain showed slightly less resistance than that fired four times, owing to the development of larger mullite crystals, which rendered the material less homogeneous. Microscopical study of thin sections showed clearly the development of mullite by repeated firings. Once-fired porcelain contained numerous quartz fragments surrounded by a glassy matrix and fine, felty, acicular crystals of mullite. In the six-fire porcelain the glassy matrix was filled with well-developed mullite crystals. Hence the examination of thin sections in conjunction with physical tests is the best method of testing the quality of finished porcelain. Felspar is capable of dissolving mullite to the extent of 9%; quantities exceeding 13% were no longer taken into solution. The solvent action of felspar on quartz has an effect on mullite formation, since mullite appeared to be less soluble in a felspathic glass rich in silica. Homogeneous fusion of quartz and felspar is complete in porcelain after the sixth fire, and mullite can then crystallise out. Experimental evidence supports the theory that alumina and silica are dissolved partly as such by felspar and partly in the form of mullite. The amorphous silica and finally the quartz also go into solution, and the highly acidified glass thus formed becomes less solvent towards mullite. Simultaneously, however, the glass becomes more viscous and crystallisation is retarded. When the viscosity is reduced (*e.g.*, by repeated firing)

the mullite readily crystallises out. Support of this theory was obtained by experimentation with a felspathic glass rich in silica, directly prepared to resemble the matrix formed in ordinary porcelain containing 25% of felspar. It follows that the quartz, depending upon its grain-size and quantity, has a determining influence upon the quantitative and qualitative development of mullite in porcelain. F. SALT.

Notes on antimony yellows. I. A. CHAMBERS and J. F. RIGG (Trans. Ceram. Soc., 1926, **25**, 101—107).—Trials were made with red lead, tin oxide, and antimony oxide, every possible mixture within the 20% limit being examined. The colours were calcined at cone 09A. The colours within the range of 40—60% of lead oxide, 20—40% of tin oxide, and 20—40% of antimony oxide gave the best results, but as underglaze colours, whether in lead or leadless glazes, they were not successful. As on-glaze colours the mixtures gave satisfactory results when mixed with four pts. of No. 8 flux (3 pts. of red lead, 2 pts. of borax, and 1 pt. of flint). The yellow colour was deepened and developed by the addition of from 1 to 5% of iron oxide. The colours were also improved by slow calcination. F. SALT.

Formulæ for slip calculations. S. R. HIND (Trans. Ceram. Soc., 1926, **25**, 108—110).—A collection of formulæ is given for use in calculations in connexion with slips of clays, various ground materials, and mixtures thereof. F. SALT.

Refractories for the pottery industry. W. EMERY (Trans. Ceram. Soc., 1926, **25**, 111—126).—A general survey is made of the requirements of refractories used in the construction of intermittent, continuous, and frit kilns, and in the manufacture of supports for ware in the kiln. F. SALT.

Tunnel kilns for burning firebricks. I. S. R. HIND (Trans. Ceram. Soc., 1926, **25**, 154—170).—The relative merits of tunnel kilns and other continuous kilns for burning firebricks are discussed. The special requirements and the chief features of design of such tunnel kilns are also outlined. F. SALT.

Some changes taking place in the low-temperature burning of Stourbridge fireclay. II. C. E. MOORE (Trans. Ceram. Coc., 1926, **25**, 127—149).—Further data are put forward in support of conclusions presented in a previous paper (cf. B., 1923, 774 A). Coefficient of expansion determinations on clay bars showed a decrease at the dehydration point, an increase at 690°, a reversal up to about 800°, followed by a continuous increase with reversals at 905° and at about 1000°. Certain small changes were also observed at approximately the same temperatures on reheating a previously fired clay, indicating that some of the changes are reversible. With rapid heating, dehydration takes place in two distinct stages; with very slow heating the water evolved in the second stage is given off progressively after the first loss is complete. The first stage represents the removal of SiO_2-water; the second, that of the Al_2O_3-water. Rehydration of fired clay was effected by prolonged treatment with a 40% caustic soda solution. The results suggest that the caustic soda solution hydrates the non-polymerised clay, decomposes it into the products normally found in the intermediate stage of the isomeric change, and then hydrates these products. Analysis of the supernatant liquor obtained by suspending powdered clays in distilled water for 24 hrs. indicates that the clays consist of a mixture of $6Al_2O_3,15SiO_2,10H_2O$ with silica and other impurities. Contraction experiments were repeated, and the three critical ranges were again observed. The complete series of changes taking place in clays on heating is: (1) loss of hygroscopic and colloidal water; (2) loss of SiO_2-water; (3) loss of Al_2O_3-water; (4) isomeric change involving decomposition and re-formation; (5) polymerisation in two or three stages. The results are all explained on the basis of the hexite–pentite theory. F. SALT.

Further note on the storage of silica refractories. W. J. REES (Trans. Ceram. Soc., 1926, **25**, 150—153).—Four types of silica bricks, varying in texture and degree of firing, were subjected to the action of water and to "weathering" tests. The results of mechanical strength tests on these and on fresh bricks indicate that highly-fired, close-textured bricks are more resistant to the action of rain, frost, etc. than less thoroughly burned bricks. Immersion for 7 days in distilled water reduced the mechanical strength of the bricks considerably, owing to partial solution of the lime bond. Rapid drying after exposure caused greater loss in strength than slow drying. F. SALT.

Refractory materials, their testing and behaviour in foundry work. E. H. SCHULZ (Stahl u. Eisen, 1926, **46**, 1667—1678).—A general discussion of the properties required in refractory materials for the various purposes of the iron industry, from the points of view, respectively, of the user and manufacturer. A plea is put forward for closer co-operation between the two, particularly in the standardisation of laboratory tests for refractories. A. B. MANNING.

Conduction of heat through [silox] powders and its dependence on pressure and conductivity of the gaseous phase. ABERDEEN and LABY.—See I.

Germanium glasses. DENNIS and LAUBENGAYER.—See VII.

PATENTS.

Glass, sand, or flint paper, emery cloth, or like abrasive. C. A. KLEIN and R. S. BROWN (E.P. 260,704, 26.8.25).—Linseed oil and paraffin or other wax is applied as a waterproofing agent to a suitable base, followed by an adhesive consisting of a drying oil with a gum, resin, or synthetic resin, and an abrasive material, the successive applications being made at temperatures above 100°. B. W. CLARKE.

Coating for [non-glare] mirror. G. F. and W. H. COLBERT (U.S.P. 1,603,936, 19.10.26. Appl., 30.4.26).—A mirror, which will reflect bright light without glare, has the reflecting surface formed of lead sulphide, produced by applying to the glass a mixture of lead acetate (1 pt.), sodium hydroxide (2 pts.), thiocarbamide (4 pts.), and water (264 pts.). Copper, nickel, and cobalt sulphides can also be employed. T. S. WHEELER.

Fusion furnace (U.S.P. 1,603,762).—See I.

IX.—BUILDING MATERIALS.

Analysis of commercial lime. T. P. BLUNT (Analsyt, 1926, **51**, 625).—Caustic lime, present in commercial lime, absorbs carbon dioxide, and may be

determined by grinding 0·4—0·5 g. of the sample with successive portions of distilled water up to 400 c.c. in all, pouring the extracts into a flask, adding the residue of the sample, closing the flask, and shaking for 30 min., rapidly filtering the contents into a known quantity of sulphuric acid, washing the filter once, and titrating the unneutralised acid. D. G. Hewer.

Effect of acids on the mechanical strength of timber. Alliott.—See I.

Vertical-retort tar for road purposes. Barash.—See III.

Distilled tar for roads. Adam and Robinson.—See III.

Patents.

Plastering walls. G. E. Heyl and O. Kunze (E.P. 260,833, 26.2.26).—A preparation for direct application to rough brick or concrete walls, thus eliminating the usual plaster layer, is made by mixing with water, *e.g.*, 40 pts. of finely disintegrated paper or wood pulp, 10 pts. of plaster of Paris or Portland cement, 20 pts. of glue, and 30 pts. of china clay, chalk, or other lubricant to promote free flow of the fibrous particles. Pigments may also be incorporated with the mixture. A. Davidson.

Manufacture of cement, lime, and the like, and kilns therefor. E. P. C. Girouard (E.P. 260,684, 7.8, 25.9, and 19.11.25).—A kiln for sintering, roasting, or calcining calcareous, argillaceous, or the like materials in dry powdered form, *e.g.*, for cement making, is formed by sinking a shaft in the natural bed available in the locality, thereby minimising external heat-radiation losses. The kiln is provided with a cooling shaft in which the air for combustion is preheated by the hot clinker, and a retort situated above the kiln for the low-temperature carbonisation of powdered coal, heated by the waste gases from the kiln, the products of carbonisation being burnt in the lower part of the kiln. B. W. Clarke.

Manufacture of quick-setting lime products. M. E. Holmes and G. J. Fink, Assrs. to National Lime Assoc. (U.S.P. 1,604,574—5, 26.10.26. Appl., [A] 16.5.23; [B] 28.5.23).—[A] Commercially pure hydrated lime is mixed with a soluble sulphate of a metal which has an at. wt. between 52 and 59. [B] A metal carbonate which readily hydrolyses is added to hydrated lime, producing a cementitious material having quick initial setting properties. B. W. Clarke.

Manufacture of quick-setting lime. J. W. Stockett, jun., Assr. to National Lime Assoc. (U.S.P. 1,604,577, 26.10.26. Appl., 17.8.25).—Quicklime is mixed with substantially completely hydrated lime in the presence of water, and the mixture is poured immediately into a mould, where reaction occurs without agitation of the mould. B. W. Clarke.

Production by carbonation and the addition of sulphates of plastic materials having an initial quick set. F. C. Mathers and R. L. Hardy, Assrs. to National Lime Assoc. (U.S.P. 1,604,576, 26.10.26. Appl., 25.7.23).—A plastic material having quick initial setting properties consists of the reaction product of carbonated hydrated lime and a soluble compound having a sulphate radical. B. W. Clarke.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Cementation of soft steel by cyanogen and cyanamide. E. Pérot (Compt. rend., 1926, **183**, 1108—1110).—The influences of time, temperature, pressure, concentration, and renewal of the gaseous atmosphere on the cementation of a steel containing 15% C in cyanogen gas have been studied. The results obtained when the gaseous atmosphere is not renewed are equivalent to those found when ethylene and methane are used in continuous circulation, and indicate that, other conditions being equal, cyanogen is a superior medium for cementation. Except so far as concerns the hypereutectoid zone, the total penetration is less with cyanogen in an atmosphere which has not been renewed than when the sample is heated in contact with tricalcium cyanamide. The micrographical structures are similar, especially at low temperatures. J. Grant.

Determination of silicon in pig iron and cast iron. H. Pinsl (Chem.-Ztg., 1926, **50**, 924—925).—The results obtained for silicon in pig iron and cast iron by the method of Rubricius (Stahl u. Eisen, 1905, **25**, 1444; cf. also B., 1905, 988) are usually a little low unless the filtrate from the first precipitate is again evaporated. The longer the heating with the strong sulphuric acid the more easily does the precipitate filter; 1·5 hrs. gentle boiling is recommended. More accurate results with only one filtration are obtained if 20 c.c. of 1 : 1 hydrochloric acid are added after the first evaporation and evaporation is repeated, followed by heating for 45 min. at 130—140°. A close-textured filter paper fitted closely into the funnel should be used for the filtration; subsequent ignition of the precipitate leaves a residue of almost pure silica, which is usually 0·05% less than the correct weight, the small amount of impurity present compensating for part of the loss of silica in the filtrate; for commercial work recovery of the traces of silica in the filtrate may be dispensed with, and no evaporation with hydrofluoric acid is necessary. The sulphuric acid method is quicker and more accurate than the usual nitric-hydrochloric acid procedure. A. R. Powell.

Equilibrium between iron, carbon, and oxygen. Reduction of iron ores, cementation, and gas occlusion of iron and steel. K. Iwasé (Sci. Rep. Tôhoku Imp. Univ., 1926, **15**, 511—529).—Existing data are used to construct a ternary equilibrium diagram of the system iron–carbon–oxygen. The use of this to determine the mechanism of the reduction of iron oxides is described. The reduction of the magnetic iron oxide by carbon takes place with the formation of the following successive phases:—

$$Fe_3O_4 \longrightarrow [Fe_3O_4] \longrightarrow [FeO] \longrightarrow [Fe] \longrightarrow [Fe_3C],$$

where the brackets indicate a solid solution based on the particular component. A small region at the iron end of the system has no gaseous phase, and here simple occlusion (*i.e.*, absorption without alteration in the composition of the gas) can take place with particular mixtures of carbon monoxide and dioxide. The mechanism of cementation in the presence or absence of oxygen is also discussed. With a particular gas mixture the

equilibrium degree of cementation is lowered with rise of temperature, but the velocity of cementation is increased. W. HUME-ROTHERY.

Nitrogenisation of steels. L. GUILLET (Compt. rend., 1926, **183**, 933—935).—Brinell tests have been carried out on a tempered case-hardened steel, and on a chrome-aluminium steel which had been nitrogenised by means of ammonia for 90 hrs. at 510°, so as to produce a nitrogenised layer 0·8 mm. thick. The latter steel had the greater initial hardness, and this was retained to a greater extent than in the case of the former, when the steels were maintained at gradually increasing temperatures (from —180° to 600°) for various periods of time. This difference was more marked when the hardness tests were carried out at 20°. After a period at the temperature of liquid air there was a distinct increase in the hardness at 20° of the nitrogenised steel, but none in the case of the other. In general, the hardness decreased as the temperature rose. J. GRANT.

Diamond cone indentation hardness tests. G. A. HANKINS (Proc. Inst. Mech. Eng., 1926, 823—842).—Accurate and reliable indentation tests can be made on hard materials with diamond cones. The indentations are easily measured and the results expressed on a rational scale. Surface lubrication had no effect on the size of the indentations. Tests made with cones of various angles gave results which were independent of the load, but dependent on the angle of the cone, the effect of the cone angle being less on hard material than on soft. The coefficient of adhesion or friction for hard steel was found to be 0·10. The corrected contact pressure $P/\pi r^2(1 + \mu \cot \theta)$ was independent of the angle of the cone from 78° to 120°, but 142° cones gave low values. Results expressed in this way were numerically the same as Brinell numbers up to 440. Above this value the Brinell numbers were lower owing to flattening of the ball. Tests with diamond pyramids had fair agreement with the cone tests up to a hardness of 500 with the latter, but fell away above this figure, and the shape of the pyramid impressions was not constant. The Rockwell test, based on depth of indentation, showed no general agreement with the pressure scales. The most suitable diamond cone has an angle of 120°, the standard load being 100 kg. The impressions must be greater than the diameter of the spherical tip. For thin sheets the point must be accurately ground to permit the use of lower loads. A fairly good surface on the specimen is desirable, but a high polish unnecessary. The 120° diamond cone is recommended for use on materials above 300 Brinell hardness, and can be used on softer materials when the values of the coefficient of adhesion are known. T. H. BURNHAM.

Comparison between Rockwell and Brinell hardness. R. C. BRUMFIELD (Trans. Amer. Soc. Steel Treat. 1926, **9**, 841—856).—Hardness determinations were made using the Rockwell hardness tester on various metals, and the results plotted against the respective Brinell hardness numbers. The Brinell test is the more sensitive except for soft metals. Equations were established for the conversion of the various hardness figures. T. H. BURNHAM.

Durometer—an instrument for testing hardness. A. SAUVEUR (Trans. Amer. Soc. Steel Treat., 1926, **9**, 929—932).—A hardened steel ball of 10 mm. diameter is allowed to drop on the specimen held at 45° and rebounds on to a horizontal graduated paper. The rebounding impulse increases with the hardness of the sample, and the scale reading of the position of fall is called the durometer number. T. H. BURNHAM.

Apparatus for measuring Hertzian hardness. ESNAULT-PELTERIE (Rev. Mét., 1926, **23**, 553—566).

Measurement of hardness by the pendulum. P. LE ROLLAND (Rev. Mét., 1926, **23**, 567—574).

Measurement of hardness by the Rockwell machine. M. NICOLLET (Rev. Mét., 1926, **23**, 575—580).

Magnetostriction. L. W. MCKEEHAN (J. Franklin Inst., 1926, **202**, 737—773).—A review of recent work.

Refractory materials, their testing and behaviour in foundry work. SCHULZ.—See VIII.

PATENTS.

Treating solid ferrous material. S. WESTBERG (E.P. 260,646, 2.6.25).—For the removal of impurities contained in iron or its alloys the material is embedded in an alkaline-earth oxide and heated to a temperature between 800° and the m.p. of the alloy in a mixture of hydrogen and an inert gas. In the elimination of sulphur, the hydrogen sulphide formed combines with the surrounding oxide, and is removed from the gaseous phase. Other materials, *e.g.*, aluminium silicide, which form stable compounds with the impurities and at the same time regenerate hydrogen, may be used to pack the metal or alloy. C. A. KING.

Manufacture of wrought iron. G. G. GEDDA (E.P. 260,789, 28.11.25).—In the ordinary process of manufacturing wrought iron, nickel-bearing material may be part of the charge of the blast furnace, thus producing pig iron containing nickel; or nickel or a nickel-iron alloy is introduced into the refining furnace together with pig iron. Cobalt may be substituted wholly or in part for nickel, and the wrought iron may contain also a small quantity of copper for the further protection from rusting. C. A. KING.

Alloy steels. H. B. KINNEAR (E.P. 260,835, 2.3.26).—An alloy steel, made by melting an ordinary carbon steel of the composition 0·2—0·6% C, 0·6—0·9% Mn, 0·2—0·6% Si, and under 0·05% of S and P, with 0·5—5% of copper, is poured into an ingot or cast, normalised by heating it at about 850° for a short time, and then slowly cooled in air. The treatment is repeated at about 550°. By varying the heat treatment it is possible to produce castings meeting different specifications from a single grade of metal. M. E. NOTTAGE.

Producing pure iron from its ore. C. E. PARSONS and S. PEACOCK, Assrs. to METAL RESEARCH CORP. (U.S.P. 1,603,710, 19.10.26. Appl., 17.10.23).—Pure iron may be produced from iron oxide free from carbonaceous material by roasting it, in the presence of a reducing gas free from sulphur, at 800—1000°. M. E. NOTTAGE.

Reduction of rare metal oxides. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,602,542, 12.10.26. Appl., 6.1.21).—A compound of the zirconium, titanium, thorium, uranium, tungsten, molybdenum,

etc. groups is heated with magnesium in the reducing atmosphere of a closed vessel, producing a refractory metal powder. H. Royal-Dawson.

Casting molten metal. A. Kadow (E.P. 260,743, 7.10.25).—Molten metal enters a partially evacuated mould through openings having a maximum width of 3/32 in. in the bottom of the mould. Increase in surface tension due to the chilling effect of the mould allows the mould when full to be lifted away from the molten metal without any further metal escaping from the narrow openings. C. A. King.

Treating aluminium to facilitate its welding and soldering. A. Kirchhof (U.S.P. 1,604,698, 26.10.26. Appl., 8.10.25. Conv., 7.3.24).—Pulverised calcium carbonate is mixed with molten aluminium from which the scum is removed. H. Royal-Dawson.

Alloys [for jets used in spinning artificial silk]. W. P. Dreaper (E.P. 260,672, 5.8.25).—An alloy of platinum and gold, containing 20—32% Pt, with or without small additions of other metals of the platinum group, is specially suitable. M. E. Nottage.

Zinc blende roasting kiln. G. Balz (U.S.P. 1,603,442, 19.10.26. Appl., 9.3.25. Conv., 21.3.24).—A zinc blende roasting kiln consists of superposed parts forming compartments, the bottom of every upper one of which forms the cover or ceiling of the next lower one, every second part in the downward direction being rotatory. The zinc blende is introduced into the uppermost compartment, and then conveyed, alternately, radially outwards and inwards through the successive compartments. It is provided with an air-heating chamber below the lowest compartments, and with means for supplying air to this chamber and discharging it into the discharge passage for the roasted zinc blende. Pipes extend radially inwards from the outside tonear, the centre of one of the intermediate hearths upon which the blende is moved radially inwards and delivered into the compartment below through a central aperture near which the pipes terminate. Means are provided for introducing cold air into these pipes. M. E. Nottage.

Sintering plant. J. E. Greenawalt (E.P. 260,895, 27.5.26).—Ore sintered in a rotating pan is dropped into a hopper beneath the pan, the bottom of which hopper consists of a perforated table on which the sinter is cooled by a current of air. The table has preferably a reciprocating motion which delivers the material on to a suitable screen for the separation of fines, and delivery of the coarser material into trucks. If necessary, the hopper is lined with cast iron, and is provided with a chimney for removing floating dust. C. A. King.

Machine for ore separation. J. F. Reilly (U.S.P. 1,603,213, 12.10.26. Appl., 18.12.23).—An oscillatory table contains a number of compartments with a porous covering. A horizontal pipe with a line of perforations on its upper side passes through each compartment. An upward flow of air is thus directed through the porous covering, and means are provided for independently controlling the supply of air to each compartment, for retarding the flow of material over the surface of the table, and for feeding granules of crushed ore over the table. M. Cook.

Vanadium compounds (E.P. 260,661).—See VII.

XI.—ELECTROTECHNICS.

Patents.

Precipitating apparatus. I. Hechenbleikner (U.S.P. 1,604,553, 26.10.26. Appl., 7.2.23).—In an apparatus for electrical precipitation, parallel vertical flues of non-conducting material and polygonal in section are situated in a chimney stack. Electrode wires are arranged centrally in each flue, with conducting supports for the upper ends of the wires, and weights at the lower ends, with a device for spacing the wires and preventing their movement. W. G. Carey.

Rendering liquids radio-active. J. G. Vaugeois (E.P. 250,935, 9.4.26. Conv., 14.4.25).—The radioactive material is separated from the liquid to be treated by an air chamber and by a porous partition permitting the passage of emanations to the liquid. The container in which the radium salt or solution is placed passes through the stopper of the vessel containing the liquid, and opens into the interior of this vessel in the free space above the liquid. W. G. Carey.

Ionic-concentration meter. E. A. Keeler, Assr. to Leeds & Northrup Co. (U.S.P. 1,604,584, 26.10.26. Appl., 7.5.20).—The method of measuring p_H values, in which a deflecting galvanometer is employed to determine the voltage between a hydrogen and a calomel electrode immersed in the solution under test, is modified by impressing on the galvanometer a voltage equal and opposite to the voltage between the electrodes, when the solution has the maximum hydrogen ion concentration. The whole scale of the galvanometer is then available for graduation in p_H values. T. S. Wheeler.

Method for and means of separating electrolytes. J. B. Speed, Assr. to Western Electric Co. (U.S.P. 1,603,298, 19.10.26. Appl., 17.12.24).—Pressure is applied to a mixture of electrolytic solutions contained in an enclosure, two walls of which are formed of porous inert conducting plates, while a potential difference, insufficient to effect electro-deposition, is applied between electrodes. The applied pressure forces a considerable portion of the liquid through the plates. J. S. G. Thomas.

[Cathode] for electrodeposition. Internat. Copperclad Co., Assees. of T. Robinson (E.P. 255,114, 4.8.25. Conv., 5.8.24).—A cathode comprises two electrically connected body portions, separated by insulating material. They are secured to a supporting bar, and are of conducting material covered with a thin film of metal different from that to be deposited on the cathode. J. S. G. Thomas.

Manufacture of electrical insulators and other moulded articles and the like. A. H. Brown and The Siluminite Insulator Co., Ltd. (E.P. 260,653, 8.7.25).—An intimate mixture of a binding material, *e.g.*, Portland cement, with less than 10% of incompletely condensed synthetic resins, together with, if desired, one or more fillers, is moulded without the addition of water, and, after removal from the mould, is subjected to heat treatment. The moulded article, after it has set and hardened, may be impregnated with a liquid synthetic resin and subjected to final heat treatment. J. S. G. Thomas.

Making electric-insulation bodies. D. M. Sutherland, jun. (U.S.P. 1,604,728, 26.10.26. Appl., 2.2.26).—A small proportion of acaroid resin and a relatively large proportion of gilsonite are added to a pulp composed of cellulose and oil soap. The mass is shaped, dried, compressed while heated under pressure, and cooled while still under pressure. J. S. G. Thomas.

Battery electrode. D. Pepper (U.S.P. 1,603,291, 19.10.26. Appl., 30.7.24).—Preformed lead material is mixed with a paste of sulphuric acid and zinc sulphate and applied by pressure to a conducting support and dried. J. S. G. Thomas.

[Depolariser for] dry batteries. Soc. Anon. le Carbone (E.P. 255,426, 1.6.26. Conv., 17.7.25. Addn. to 228,872; cf. B., 1926, 550).—The depolariser consists of powdered charcoal distributed throughout an immobilised material, and covered by a film rendered impermeable to liquids prior to its incorporation in the mass containing the electrolyte by being mixed with paraffin oil. J. S. G. Thomas.

XII.—FATS; OILS; WAXES.

Plant for fat extraction. L. J. Simon and J. W. Hinchley (J.S.C.I., 1926, **45**, 252—259 t).—Extraction of oil-seeds in a 3—4 ton vessel leads to loss of time due to the long time occupied by the flow of solvent through a large mass of meal, involves distillation of weak solutions of oil, and further involves much labour and loss of time between each extraction during operations of discharging and re-charging with crushed meal. A long period of steaming the meal for removal of solvent results in a high percentage of moisture being present, and the length of time during which meal and oil are subjected to heat and moisture may lead to deterioration of the oil. These objections are overcome in a plant holding only 4 cwt. of material at one time, though the output is 3—4 tons in 8 hrs. Total time of extraction is about 30 min., and total time of "steaming off" of the meal to remove solvent is 4—6 min., these results being achieved by preheating the meal to the temperature of steam, by the fact of steam having to percolate through only a few inches of material, and all meal being in contact with solvent for the same length of time. The distillation of the oil solution is taking place continuously, only solvent well saturated with oil entering the stills. In the plant described only about 1 ton of solvent is needed to treat 3 tons of meal, as against 7 tons when 3 tons of meal are treated at one charge. The cage holding the meal consists of a perforated drum carried on a hollow shaft through which the solvent and steam are allowed to enter—the meal being charged into the cage by removal and replacement of one of the end plates—and the cage is inserted into a cylinder carrying the gear for rotating it. Each machine carries three such cylinders with their cages, each operated separately. After the operation of charging and discharging, automatic hydraulically-controlled valves operated by means of a timed "cam" shaft which itself determines the course of all the operations from the entry of the solvent until the extraction and "steaming off" are completed. This timed shaft makes one complete revolution in 32 min., by which time an extraction is completed and the cage containing the meal is ready for removal. The solution tanks and solutions are collectively dealt with, the complete plant having in addition the usual condenser and separator, solvent tank, six solution tanks, one solvent measuring tank, and a distilling apparatus. The operation of the machine consists of (1) a preliminary treatment of the dried meal with solvent vapour; (2) a washing of the material with a concentrated solution of oil and solvent to obtain a rich solution for distillation; (3) a second treatment with solution which is used for the next charge for operation 2; (4) a third treatment, which is used in the next charge for operation 3; (5) a final treatment with pure solvent; (6) a drying period, in which most of the solvent is expelled from the meal by centripetal force, the material being warmed by indirect steam; and (7) steaming off with direct steam to remove the last traces of solvent from the meal. This method of working ensures a highly concentrated oil solution for distillation in a regulating still which, while continuously evaporating the solvent by means of a closed steam coil, converts the intermittent flow which it receives into a regular stream, which enters a set of six small stills coupled in two equal sets in parallel. During periods 1 to 5 the cage is rotating slowly, ensuring constant agitation of the meal, and in periods 6 and 7 rapidly so as to form the meal into a cylinder with a wall of even thickness which allows steam to pass through quickly, ensuring effective removal of the last traces of solvent. Only the final flush is clean solvent, and the total solvent used for each charge is only 16—20 gallons. The claims for the system are (1) more complete extraction, (2) less use of solvent, (3) lower steam consumption, and (4) dryer meal after extraction. (Cf. E.P. 255,923; *infra*.) H. M. Langton.

Phytosterols of rice-bran fat. F. P. Nabenhauer and R. J. Anderson (J. Amer. Chem. Soc., 1926, **48**, 2972—2976).—Extraction of rice-bran with light petroleum yielded about 10% of an oil, consisting largely of free fatty acids. The unsaponif. matter (about 5%) contains myricyl alcohol (cf. Jamieson, J. Oil Fat Ind., 1926, **3**, 256), dihydrositosterol (Anderson, Anderson, and Nabenhauer, B., 1924, 691, 729), stigmasterol (Windhaus and Hauth, A., 1907, i. 129), and a phytosterol, probably not the homogenous sitosterol of Weinhagen (A., 1918, i, 56). No substance resembling the latter's saturated hydrocarbon (*loc. cit.*) was identified. Some oily constituents (b.p. 100—260°/1mm.) were not fully identified. F. G. Willson.

Phytosterols of corn [maize] oil. R. J. Anderson and R. L. Shriner (J. Amer. Chem. Soc., 1926, **48**, 2976—2986).—The crystalline unsaponif. matter present in maize oil is a mixture of dihydrositosterol, stigmasterol (cf. Anderson and Moore, B., 1923, 937 a), and three isomerides, α-, β-, and *γ-sitosterol*, of which only the last was obtained pure. The crystals have the formula $C_{27}H_{45}OH,H_2O$, m.p. 145—146°, $[\alpha]_D$, in chloroform solution, $-42\cdot43°$; *γ-sitosteryl acetate* has m.p. 143—144°, $[\alpha]_D$, in chloroform solution, $-46\cdot09°$. *Dibromo-γ-sitosteryl acetate*, m.p. 136—137°; *dihydro-γ-sitosterol*, from the parent hydrocarbon by reduction with hydrogen in presence of platinum-black, m.p. 144—145°, $[\alpha]_D$, in chloroform solution, $+17\cdot82°$, and *dihydro-γ-sitosteryl acetate*, m.p. 143°, $[\alpha]_D$, in chloroform solution, $+8\cdot98°$,

are also described. γ-Sitosterol is possibly identical with the *p*-phytosterol of Likiernik (A., 1891, 606). It appears that the sitosterol previously described by Anderson and Moore (*loc. cit.*) is a complex mixture of isomerides. F. G. Willson.

Phytosterols of wheat-germ oil. R. J. Anderson, R. L. Shriner, and G. O. Burr (J. Amer. Chem. Soc., 1926, **48**, 2987—2996).—The crystalline sterols in American-grown wheat-germ oil contain no pure homogeneous sitosterol, but constitute a mixture of α-, β-, and γ-sterols (cf. preceding abstract; Burian, A., 1898, i, 72; Ritter, A., 1902, i, 446) and dihydrositosterol, with possibly other constitutents. As with maize oil (cf. preceding abstract) only γ-sitosterol could be obtained pure. American-grown wheat appears to contain different sterols from wheat grown in Europe. F. G. Willson.

Methods of saponification in the light of modern saponification theory. C. Bergell (Z. deuts. Oel- u. Fett-Ind., 1926, **46**, 737—738, 753—754, 769—770).—The first two sections of this paper deal mainly with the empirical development of soap-boiling processes during the last century. It is then shown that the velocity of saponification $= v = KC_1C_2$, where C_1 and C_2 are the concentrations of esters and alkali respectively, and since C_1 and C_2 decrease in geometric progression, v also decreases. This equation would hold for a molecularly homogeneous solution, but in the case of emulsions in soap boiling a modification results from the fact that the surface of the fat globules does not vary directly with the decrease of weight of fat as the saponification proceeds. Experiments are then described in which specimens of coconut oil and tallow are saponified at varying temperatures, and with varying amounts of alkali in excess of that theoretically required, the results of acid value determinations being tabulated and plotted. When the amount of saponification per unit time is plotted against time a minimum occurs in the first third of the curve in each case, this being the point at which the lye in oil emulsion changes over to an emulsion of oil in lye, the actual position varying with conditions of temperature, concentration of lye, and the kind of fat. In one case separation occurred half-way through the experiment, and the rate of saponification fell off steeply. The general conclusion is that the maintenance of a good emulsion is more important than a high saponification temperature, and the author is of opinion that the cold saponification process merits much wider application in the soap industry. E. Holmes.

Saponification of olive oil. G. I. Finch and A. Karim (J.S.C.I., 1926, **45**, 469—472 T).—Olive oil was saponified with equivalent quantities of aqueous caustic soda solutions of various strengths under various conditions of stirring. An optimum alkali concentration of 29·25% was found, leading, with suitable stirring conditions, to a 98% saponification within 30 min.

Acetin and dichromate methods [for glycerin analysis]. O. Sachs and K. Riemer (Z. deuts. Oel- u. Fett-Ind., 1926, **46**, 739—740).—Following the communication of Prager (B., 1926, 923), the authors tabulate the results of numerous analyses of glycerin by these two processes, the differences in no case being in excess of 0·11%. They consider the acetin process equal in accuracy to the dichromate process, over which it has the advantage of quicker manipulation, and are strongly in favour of its retention. H. M. Langton.

Rate of polymerisation of perilla oil. M. Toch (J. Oil Colour Chem. Assoc., 1926, **9**, 309—318).—The thickening of heated perilla oil was studied both in the laboratory and under factory conditions, changes in sp. gr., refractive index, acid value, iodine value, and mol. wt. being recorded. Very little change occurs at low temperatures, but at 304° the iodine value decreases rapidly, while the remaining constants show a corresponding increase, the rates of these changes falling off with the time the oil is maintained at 304°. The changes are compared with the very similar ones for linseed oil, perilla oil being shown to lag at lower temperatures, but to polymerise faster than linseed oil at the high temperature. The mechanism of oil polymerisation is briefly discussed. S. S. Woolf.

Iodine value of paprika oil. L. C. Mitchell (J. Assoc. Off. Agric. Chem., 1926, **9**, 477—482).—The oil extracted from paprika is unstable, and special precautions are necessary to prevent oxidation, or low iodine values will be obtained. A simplified method, which yields results in close agreement with the anhydrous ether method, and is also much quicker, consists in extraction with chloroform, and determination of the quantity of iodine absorbed and of the weight of oil used on aliquot portions of the chloroform solution. F. R. Ennos.

Tertiary bromine–iodine values of drying oils. W. Vaubel (Farben-Ztg., 1926, **32**, 514).—To the primary and secondary bromine–iodine values previously described by the author (cf. B., 1926, 887) a tertiary value is added, obtained by the action of excess of bromine for three days (in the secondary value the excess is immediately determined). The calculated bromine–iodine values for a number of drying and non-drying oils, and of oil films at various ages, are tabulated. The tertiary values, which are much in excess of previous highest iodine values, sometimes rise on the ageing of oil films, whereas primary and secondary values generally fall. The significance of this new value and its effect on the oil analyses previously given (*ibid.*) are discussed. S. S. Woolf.

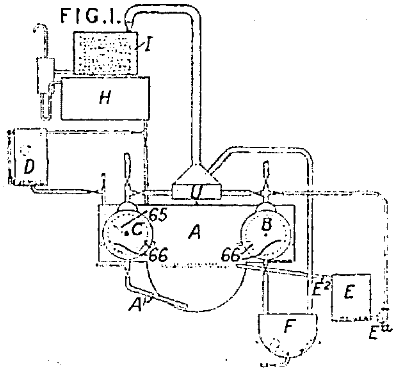
Importance of soya-bean oil for softening and increasing the stretch of cold-vulcanised rubber. Ditmar.—See XIV.

Patents

Extraction of oils, fats, waxes, greases, and the like. L. J. and A. Simon, and Simon Bros. (Engineers), Ltd. (E.P. 255,923, 20.4.25 and 17.6.25).—Improved results are achieved in a machine which treats small quantities of meal at a time, leading to a high rate of solution of fatty oil and thorough removal of solvent from meal. Solvent is conveyed by large entry pipes on to a few inches depth of meal contained in a rotating cage, whereby continuous agitation with solvent occurs, and the amount of liquor left in the meal on drainage is a minimum. The cage consists of two end plates on a hollow, perforated, central shaft with gauzes, and/or with filter cloth fixed to the periphery of the plates as well as around the shaft. The charged cage is placed in

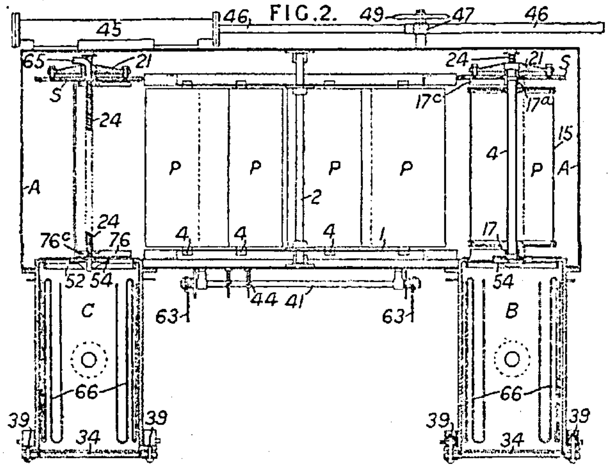
a cylinder carrying the gear for rotating it, several such cylinders being contained in each machine. Coupled with the machine are a large tank A, which has a constant supply of solvent, and two smaller tanks, B, C, one for preliminary extraction, and one for the final flush and steam, and these adjoin and open into A, from which

FIG. 1.



they can be isolated. A cam-operated hydraulic contrivance controls the whole cycle of operations, at the start of which a charged cage is placed in the small tank B, in which it is subject to a preliminary solvent flush, transferred to the large tank A, capable of holding several such cages, and subsequently to the small tank C

FIG. 2.



for giving the material a final flush of solvent and for steaming off solvent from the meal. Means are provided for supplying solvent or steam to the centre of the cages through the perforated shaft thereof. The tanks are provided with the usual coils, and stills, condensers, and solvent and solution storage tanks form part of the installation. (Cf. Simon and Hinchley, *supra.*)

H. M. LANGTON.

Apparatus for cooling oil vapours (E.P. 240,415).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Noxious solvents and thinners used in paints and adhesives. A. BEYTHIEN (Farben-Ztg., 1926, **32**, 567—569).—The harmful effects of the vapours arising in industrial operations involving the use of special paints and adhesives are indicated, and the ingredients of several technical preparations are discussed from this viewpoint.

S. S. WOOLF.

Colloid-chemical studies on chrome yellow. L. BOCK (Farben-Ztg., 1926, **32**, 459—460).—The improvement in the tone of pale chrome yellows by co-precipitation with colloidally-dispersed inorganic substances, *e.g.*, aluminium hydroxide, lead sulphate, aluminium phosphate, lead arsenite, etc., is also effected by the use of organic substances such as animal or vegetable glues. Whilst these protective colloids maintain or increase the degree of dispersion of the precipitated pigment, the latter is still subject to the same darkening under the action of light as is pure (normal) lead chromate.

S. S. WOOLF.

Cobalt blues and allied colours. J. J. FOX (J. Oil Colour Chem. Assoc., 1926, **9**, 304—308).—An account of the nature, details of preparation, uses, and methods of analysis of various cobalt pigments.

S. S. WOOLF.

U.S. Government master specification for turpentine (gum spirits of turpentine and steam-distilled wood turpentine) (U.S. Bureau of Standards, 1926, Circular No. 86, 1—10).—The requirements for either type of turpentine are as follows:—$d^{15.5}_{15.5}$ 0·860—0·875; n^{20}_{D} 1·465—1·478; after polymerisation with 38N-sulphuric acid, not more than 2% of a viscous, straw- or darker-coloured residue, having n^{20}_{D} 1·500 (minimum) shall be obtained; on distillation under specified conditions, the initial b.p./760 mm. shall be 150—160°, whilst at least 90% shall distil below 170°/760 mm. Details of apparatus and methods for sampling and testing are given. The specification does not cover destructively distilled wood turpentine.

S. S. WOOLF.

Fluorescence- and capillary-analysis of resins. H. WOLFF and W. TOELDTE (Farben-Ztg., 1926, **32**, 294—296).—To avoid errors in fluorescence analysis of resins due to the condition of the latter, it is proposed to examine, under the analytical quartz lamp, the "adsorption pictures" obtained under standard conditions by Stock's method of capillary analysis (cf. B., 1926, 679). A full description of the characteristic phenomena exhibited by various resins and resin mixtures when treated in this way is given.

S. S. WOOLF.

Industrial resinates. R. UZAC (Rev. gén. Colloid., 1926, **4**, 257—268; cf. A., 1925, i, 1295).—Resin soaps contain an acid resinate which can be extracted by means of toluene. In the gumming of paper an aluminium resinate, containing free resin, is formed by addition of aluminium sulphate to the resin. Resin alone fixes on the fibre, but the gum is not permanent. On the other hand, treatment of the paper with alcohol causes a loss in adhesive power, due to dissolution of free resin. It is shown that a colloidal complex is formed between aluminium resinate and resin, and it is suggested that, since cellulose readily adsorbs aluminium hydroxide, a colloidal complex of aluminium resinate, resin, and cellulose is formed at the surface of the paper. A study is made of the viscosities of preparations of the resinates of lead, zinc, calcium, and manganese, and it is concluded that colloidal complexes of basic resinates and free resin are formed, rather than complexes of metallic oxides and resin.

E. S. HEDGES.

PATENTS.

Preparation of paint filler. T. G. GAUDRY, Assr. to A. HOOLAHAN (U.S.P. 1,604,904, 26.10.26. Appl.,

12.5.23).—An aqueous solution of oxalic acid, alum, and washing soda is added to a methylated spirit solution of rosin under steam pressure, and after successive additions of aqueous solutions of potassium hydroxide, zinc sulphate, lead acetate, gum arabic, sodium hydroxide, and sodium silicate, the whole is stirred in a large volume of water. S. S. Woolf.

Treating paints [to improve flexibility]. R. Mezger, Assr. to P. Lechler (U.S.P. 1,604,930, 26.10.26. Appl., 7.9.23. Conv., 1.8.22).—Paints containing non-aqueous vehicles are rendered more flexible on hardening by adding a small amount of a soluble salt of an iron-group metal, and precipitating the insoluble hydroxide in the paint by adding an alkali hydroxide, the amount of water so introduced being less than the amount of non-aqueous vehicle. S. S. Woolf.

Making ["non-breaking"] varnish oil. A. Schwarcman, Assr. to Spencer Kellogg & Sons, Inc. (U.S.P. 1,604,425, 26.10.26. Appl., 27.6.25. Cf. U.S.P. 1,407,952; B., 1922, 301 a).—Raw linseed oil is emulsified with a solution of zinc sulphate (0·1% Zn on the oil treated), and an equivalent quantity of sodium hydroxide is added. The mixture is heated under reduced pressure to evaporate the water present and filtered. The product can be heated to 260° without clouding ("breaking"). Other freshly-precipitated, hydrated oxides, which do not catalyse the drying of the oil, *e.g.*, hydrated alumina or lime, can also be employed. T. S. Wheeler.

Method and apparatus for removing large-sized particles and aggregates from certain finely divided powders and the like (E.P. 260,741).—See I.

Manufacture of articles having surfaces capable of producing diffraction colour effects (E.P. 260,669).—See I.

Separators for removing entrained oil from hot vapours (E.P. 260,899).—See I.

Manufacture of electrical insulators and other moulded articles (E.P. 260,653).—See XI.

Photographic medium [or varnish] (U.S.P. 1,604,674).—See XXI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Smoked sheet rubber prepared with dinitro-*o*-cresol. H. P. Stevens (Bull. Rubber Growers' Assoc., 1926, **8**, 594—596).—Experiments with smoked sheet rubber in the preparation of which 0·2 g. or 0·5 g. of 3:5-dinitro-*o*-cresol was introduced with the coagulant into 3000 c.c. of latex, confirm the earlier finding with unsmoked sheet that this substance prevents the formation of mould, and does not appreciably influence the vulcanising properties of the rubber. D. F. Twiss.

Importance of soya-bean oil for softening and increasing the stretch of cold-vulcanised rubber. R. Ditmar (Gummi-Ztg., 1926, **41**, 535—536).—"Dipped" goods containing up to 20% of soya-bean oil (calculated on the rubber) and vulcanised by immersion in a solution of sulphur chloride in carbon disulphide show a high stretch. If vulcanised in sulphur chloride vapour, or if containing a higher proportion of the oil, the products deteriorate rapidly. D. F. Twiss.

Preparation of a standard plantation rubber. F. B. Jones (Trans. Inst. Rubber Ind., 1926, **2**, 180—200).—The ideal plantation rubber should be as uniform as possible in plasticity and rate of vulcanisation, in order to reduce the tendency to pre-vulcanisation and "scorching" during manufacture; relatively slow vulcanisation is, however, an advantage. For the increase of uniformity, latex should be bulked as far as possible, and the conditions of coagulation and after-treatment carefully standardised. D. F. Twiss.

Connexion between constitution and accelerator action of diarylthioureas and diarylguanidines. W. J. S. Naunton (J.S.C.I., 1926, **45**, 376—384 t).—For the study of the connexion between chemical constitution and vulcanisation accelerator action the substances to be tested should be free from impurities which could possibly enhance the action (*i.e.*, that could give the "two-accelerator effect"), and the greatest care is necessary in making the vulcanisation tests. Standardisation of every operation, from milling of the raw rubber to temperature control of the tensile tests, is essential. The work necessitated the characterisation of the hitherto undescribed *di*-m-*tolylguanidine*, m.p. 116—117°, *di*-o-*xylylguanidine*, m.p. 247—248°, *di*-p-*xylylguanidine*, m.p. 165—166°, *di*-β-*naphthylguanidine*, m.p. 200—200·5°, *di*-p-*nitrophenylguanidine*, m.p. 222°, and *tetramethyldiaminodiphenylguanidine*, m.p. 162—163°. It was found both in the diarylthiourea and diarylguanidine series that groups which tend to increase the aromatic nature of the molecule tend to increase the accelerator activity, whereas groups which intensify the aromatic nature (*i.e.*, electro-negative groups) decrease the potency; thus the dinitrodiphenylguanidines have practically no accelerator action. Hydroxyl groups decrease the activity in a zinc oxide mix, but the metallic salts of such compounds are more active in a pure rubber-sulphur mix than the parent substances. The nitro-derivatives are capable of vulcanising rubber in the presence of litharge and the absence of sulphur, but have no interest as accelerators in either zinc oxide or litharge mixes. The basic derivatives, especially in the thiourea series, exhibit marked superiority over the parent substances when used in mixes containing substitute, and also confer better ageing properties, as demonstrated by rapid ageing at 70° upon vulcanisates containing them. The introduction of a second thiourea group in the same molecule results in a less active accelerator instead of a more active; the increased insolubility of such substances may, however, partly account for this effect. A simple attachment for recording automatically the loads at given extensions of the dumb-bell in the testing machine is also described.

Analysis of vulcanised rubber. Direct determination of free carbon. E. Kahane (Caoutchouc et Gutta-Percha, 1926, **23**, 13331—13334).—Cautiously treat 1 g. of the rubber in small pieces with 6—8 c.c. of fuming nitric acid; after the initial vigorous action, boil the mixture for 5 min., dilute to 50 c.c., and keep hot until decantation is possible. Decant into a Gooch crucible, the bottom of which is covered by a disc of filter paper, wash with warm dilute nitric acid, and then repeatedly with a mixture of 20 c.c. of boiling

water, 2 c.c. of acetic acid, and 5 c.c. of concentrated ammonia solution. Drain with the aid of suction until the solid in the crucible can be detached and returned (together with the paper) to the beaker. Repeat the previous treatment, but using 5 c.c. of fuming nitric acid and washing with dilute nitric acid and with boiling water after the ammoniacal solution. Dry 2 hrs. at 120° and weigh with a similarly-treated disc of filter paper on the other pan of the balance. Finally oxidise the carbon at a dull red heat. The loss in weight represents 110% of the content of free carbon.

D. F. Twiss.

Physical properties of caoutchouc. M. Le Blanc and M. Kröger (Kolloid Z., 1926, **40**, 143—144).—Explanatory in reply to van Rossem and van der Meyden (cf. B., 1926, 502). E. S. Hedges.

Patents.

Accelerator for the vulcanisation of rubber and method of making same. L. B. Sebrell, Assr. to Goodyear Tire and Rubber Co. (U.S.P. 1,604,199, 26.10.26. Appl., 4.4.24).—A mercaptobenzothiazole is produced by heating a mixture of an aryldithiocarbamate with sulphur to between 100° and 400° under a pressure of 100—3000 lb./sq. in. D. F. Twiss.

Method of vulcanising caoutchouc and product obtained thereby. C. M. Carson, Assr. to Goodyear Tire and Rubber Co. (U.S.P. 1,603,317, 19.10.26. Appl., 15.2.24).—Rubber is mixed with a vulcanising agent and an aminothiophenol and then vulcanised.

D. F. Twiss.

Rubber-vulcanisation accelerator. W. Scott, Assr. to Rubber Service Laboratories Co. (U.S.P. 1,606,321, 9.11.26. Appl., 17.12.24).—A mixture of plasticised rubber and guanidine polysulphide is vulcanised. S. S. Woolf.

Liner for rubberised material and method of treating same. H. A. Endres, Assr. to Goodyear Tire & Rubber Co. (U.S.P. 1,603,334, 19.10.26. Appl., 4.11.22).—A liner is treated with a composition containing free sulphur and a material capable of wetting the sulphur. D. F. Twiss.

Making a rubberised fibre composition. J. M. Gillet, Assr. to Goodyear Tire & Rubber Co. (U.S.P. 1,603,335, 19.10.26. Appl., 31.7.22).—Rubber is dissolved in a mixture of toluene and alcohol above a temperature y indicated by the equation : $7y - 33x = -563$, where x represents the percentage of alcohol; precipitation of the rubber is then effected by reducing the temperature of the solution below the level represented by the equation $7y - 33x = -605$.

D. F. Twiss.

Manufacturing a rubberised fibre composition. J. T. Charleson, Assr. to Goodyear Tire & Rubber Co. (U.S.P. 1,603,318, 19.10.26. Appl., 31.7.22).—A solution of rubber containing a quantity of fibres is heated; the depolymerised rubber is subsequently precipitated on to the fibres, which are then pressed and vulcanised. D. F. Twiss.

Concentrating [rubber] latex. K.D.P., Ltd. (E.P. 244,727, 13.11.25. Conv., 20.12.24. Cf. E.P. 243,016; B., 1926, 956).—In the concentration of heated latex (stabilised or vulcanised, if desired) by exposure to a current of air in a rotating drum, an unsupported cylinder, present inside the rotating drum and rolling continuously in the latex, aids the exposure of the latex to the drying gases, and also ensures uniformity in the concentrated product. D. F. Twiss.

Impregnating leather and other materials with rubber. N. J. S. Nunn (E.P. 260,652, 3.7.25).—Leather, fabrics, or paper can be impregnated with a solution containing, *e.g.*, rubber, benzene, naphtha, carbon tetrachloride, isoprene, and acetone at 63° ; the drained and dried material is then subjected to any desired finishing process. Chrome-tanned leathers treated in this way are improved in their waterproof qualities, durability, and pliability. D. F. Twiss.

Method and apparatus for removing large-sized particles and aggregates from certain finely-divided powders and the like. (E.P. 260,741).—See I.

XV.—LEATHER; GLUE.

Standardisation of hide powder [for tannin analysis]. II. H. G. Bennett (J. Soc. Leather Trades' Chem., 1926, **10**, 355—374. Cf. J.S.C.I., 1907, 455).—Recently suggested criteria for judging hide powder, viz., the alkalinity of the ash (B., 1925, 558) and the p_H value (B., 1926, 454), do not distinguish between powders B8 and B11, which were so widely different in use, and therefore the tests cannot be useful in revealing similar future differences. The adoption of an official adsorption test is recommended using the different hide powders under examination in conjunction with a standard solution of gallic acid. The author's previous method (Collegium, 1917, 56, Lon. Edn.) gives comparative adsorptions for B3, B4, B8, and B11 as 100, 92, 104, and 96, respectively, thus indicating those powders showing bad adsorption. To meet criticisms, the author has modified the adsorption test. A 0·6% solution of gallic acid is used, and a tannin analysis done on this solution. The adsorption ratio, A, is the ratio of the gallic acid adsorbed by the powder to the unadsorbed portion. An alternative method is to carry out the test with two solutions containing 0·6 and 0·7% respectively, churn with the chromed powder for 30 min., and use the equation $y = ac + b$, where y = amount adsorbed and c the amount unadsorbed, to find the constants a and b, and thus determine the adsorption ratio for the standard solution. The current official hide powder B13 is very variable in quality, and it is suggested that the calculated mean variations of the adsorption ratio, the test for soluble matter, and the percentage of mineral ash should also be used as official criteria for hide powders. The adsorption ratio and the mean variations of this and other determinations should be used by the Society of Leather Trades' Chemists in the adoption of an official hide powder, and also by the hide powder manufacturers in controlling the manufacture. The " relative specific surface " of B8 powder is practically the same now as it was two years ago, so that its absorbency has not changed. The adsorption ratio for B8, a satisfactory powder, is $1·30 \pm 0·04$. $A = 1·30$ as a minimum, and the mean variation $\pm 0·04$ as a maximum may be considered the desiderata of a good hide powder. D. Woodroffe.

Characteristics of hide- and sinew-collagen and their behaviour towards ferments. W. S. SSADIKOV (Collegium, 1926, 512—518).—Alkaline solutions of tryptase or collagenase-free trypsin hydrolyse fibrin into amino-acids, but not collagen. Acid solutions of α-glutinase-free pepsin dissolve collagen, forming non-glutinous peptones, without formation of amino-acids. Papayotin dissolved collagen in neutral solution at 80°, forming decomposition products similar to amino-acids. Collagenase, free from trypsin, peptises collagen in alkaline solution without forming amino-acids. α-Glutinase, in acid solution, converts collagen into a jelly-like glutin, resistant to acids and α-glutinase. β-Glutinase, in alkaline solution, transforms collagen into a jelly, which is resistant to the action of caustic soda and β-glutinase. The above properties are general for collagens of different origins and different preliminary treatments. A sample of sinew-collagen treated with a 0·25% solution of caustic soda, was preserved in alcohol for 20 years, and was found to differ from fresh sinew-collagen. It was not attacked by an alkaline solution of trypsin and collagenase, an acid solution containing 5% of pepsin, or by papayotin. It required an 8% solution of pepsin to decompose it. Animal hide differs from hide powder and sinew in that it is almost completely dissolved by commercial pepsin. Commercial trypsin has no decomposing action, whilst sinew is partially attacked. Papayotin has no action on hide. A table is given showing the comparative behaviour of sinew, hide, and fibrin towards various fractions of the ferments obtained by treating the latter with charcoal or kaolin and determining the action of the absorbent and the clear liquor respectively. Hide is much more resistant to enzymes than sinew. Collagenase has no action on hide either in acid or alkaline solution, and pepsin treated with kaolin has only a slight swelling action. After calf-skin has been finely divided and treated with weak caustic soda solution, like sinew, the product, hide collagen, can be dissolved by all the above-named enzymes, viz., trypsin, pancreatin, pepsin, collagenase. The differences between sinew- and hide-collagen are apparent only, being due to differences in preliminary treatment, *e.g.*, hydration, maceration. Experiments have shown that bates of the "Oropon" type contain tryptase and collagenase, which do not dissolve the hide provided the latter has preserved its original properties. Hide powder, though only slightly changed chemically, is definitely attacked by "Oropon" bates.

D. WOODROFFE.

Sulphato-hydroxo-chromi-collagen compound. K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1926, **21**, 559—570).—Samples of delimed calf-skin were pickled with sulphuric acid and sodium sulphate, also one-bath chrome tanned and washed, respectively. Pieces of each were cut from corresponding parts of the samples and analysed, and portions shaken with distilled water. The diffused acid was neutralised with $0 \cdot 1N$-sodium hydroxide solution at frequent intervals. Equilibrium was established after 48 hrs. treatment, and the pickled stock had then yielded up all its acid. From the titration figures of alkali consumed, the rates of hydrolysis of the collagen–sulphuric acid compound in pickled and tanned stock were shown to differ. If the total acid sulphate in chrome leather existed as protein sulphate, the rate of hydrolysis should have been the same as for pickled pelt. Chrome leather which had been washed for 48 hrs. showed the acidity of the chromium–collagen compound to be 32·9 and 31·4%. The p_H values of the solutions obtained by treating 5 g. of the pickled skin and chromed skin respectively with 200 c.c. of water for 24 hrs. were 2·81 and 3·71. As the concentration of the chrome tanning liquor increased, so the acidity of the neutralised tanned leather increased at first, reached a maximum, and then diminished slightly. The increase in acidity follows from $Cr[(H_2O)_6]^{+++} \rightleftharpoons Cr[X(H_2O)_5]^{++}$ where the formation of a heterogeneously substituted internal sphere is favoured by increase in concentration. The decrease with higher concentrations is probably due to the transference of cationic chromium complexes into the anionic state. Portions of hide powder were tanned for 48 hrs. with a 63% chrome liquor containing sodium sulphate, and the samples washed to remove protein sulphate; the acidity of the neutralised product increased from 37·9 to 49·1% with increasing amount of sodium sulphate, whereas the acidity of cationic chromium complexes fixed by permutit from the same chrome liquors showed an increase with small addition of sodium sulphate and a decrease with subsequent larger additions. Evidently electro-chemical changes take place in the chromium salt in the presence of large amounts of sodium sulphate. Present methods of controlling chrome tanning liquors, *e.g.*, p_H figure and precipitation figure, have only a limited application, the composition of the internal sphere being the important factor. Its acidity may show variation from 20—50%.

D. WOODROFFE.

Action of neutral salts on hide protein. G. D. MCLAUGHLIN and E. R. THEIS (Collegium, 1926, 431—436).—Fresh corium and hide powder prepared therefrom were treated with saturated solutions of sodium chloride and sodium sulphate respectively. Fresh corium was dissolved equally well by each solution, but sodium chloride proved a better antiseptic than sodium sulphate. In the absence of bacteria, hide powder prepared from fresh corium is dissolved to a less extent by sodium sulphate than by water or sodium chloride. The latter exercises a greater hydrolytic effect than water. The researches of others (cf. B., 1925, 292; 1926, 23, 503) have shown that the difference between the solvent action of sodium sulphate and chloride, respectively, increases with increasing change in the hide proteins, *e.g.*, in hide powder for analysis and in gelatin. When a tissue or an albuminous substance contains more than one protein, and when one or more of these are characterised by different solubilities in salt solutions, then the nitrogenous compounds dissolved by the latter must differ. The effect of neutral salts depends on the non-coagulable portion or on the hydrolysed proteins in the hide corium.

D. WOODROFFE.

Comparison of the tanning action of extracts and crude tanning materials. P. PAVLOVITSCH (Collegium, 1926, 441—445).—Comparative analytical tests have been made on pieces of hide which had been coloured in the same weak tan liquors and subsequently tanned in ordinary "layers," in "layers" dusted with sterilised

materials, in clear extract liquors, and in turbid extract liquors respectively. There was no difference between the results obtained from clear or turbid liquors nor between sterilised and natural dusting materials. Extract liquors were not inferior to "layers." There was no evidence to support the view that enzymes exercise a favourable effect on the tannage (cf. B., 1923, 367). D. Woodroffe.

Comparison of boric and hydrochloric acids in the determination of nitrogen in leather. [Report of Committee of American Leather Chemists' Association.] R. W. Frey and R. M. Hann (J. Amer. Leather Chem. Assoc., 1926, **21**, 583—587).—Various samples of chrome- and vegetable-tanned leathers have been used for comparative determinations of the nitrogen content by Kjeldahl method, distilling the ammonia into $N/5$-hydrochloric acid and 4·5% boric acid respectively. The results agreed very well. In some cases the boric acid yielded slightly higher figures. The adoption of boric acid for collecting the ammonia is recommended. D. Woodroffe.

Absorption of acid and basic dyes by cationic and anionic chrome-tanned hide powder. K. H. Gustavson (Collegium, 1926, 437—441).—Cationic chromium appears to combine with the acid groups in collagen, whereas the anionic chromium combines particularly with the basic groups. Samples of hide powder separately tanned with each kind were tested with acid and basic dyestuffs respectively. Acid dyes are taken up by hide protein on the acid side of the isoelectric point only, whilst basic dyes are absorbed on the alkaline side only. Acid dyes showed a much greater affinity for hide powder tanned with cationic chromium than for anionic chrome-tanned powder or for powder tanned with anionic and cationic chromium, whereas basic dyes were absorbed more readily by anionic chrome-tanned powders. Vegetable-tanned hide powder behaves towards acid dyestuffs in the same way as powder tanned with anionic chromium compounds, which confirms Procter's view ("Principles of Leather Manufacture," 1922, 491) that tanning is mainly a function of the condition of the basic groups in the collagen. The dyeing of unmordanted chrome leather with acid dyes depends on the hydrogen-ion concentration and on the condition of the inactive protein groups. The different behaviour of one- and two-bath chrome-tanned leathers towards acid dyes can be attributed to the dual mechanism of chrome tannage; two-bath leather comprises anionic chromium complexes only. D. Woodroffe.

Noxious solvents and thinners used in paints and adhesives. Beythien.—See XIII.

Patents.

Method of moulding and hardening algin-containing material. C. C. Loomis and A. L. Kennedy, Assrs. to Kelp Products Co. (U.S.P. 1,603,783, 19.10.26. Appl., 17.8.21).—The algin in the material is converted into an insoluble alginate, and a relatively small amount of softening agent is added (insufficient to bring the total moisture content above 25%). The softened material is moulded by pressure, and hardened by the removal of sufficient water. S. S. Woolf.

Preparation of japanned leather goods. G. E. Haldinstein (E.P. 260,803, 23.12.25).

Impregnating leather with rubber (E.P. 260,652). —See XIV.

XVI.—AGRICULTURE.

Stimulation of plant growth by potassium iodide. O. Loew (Z. Pflanz. Düng., 1926, **A7**, 233—234).—In cylinder experiments with barley, using increasing dressings of potassium iodide, a maximum yield was obtained with a dressing equivalent to 500 g./hectare. The increase over the control for this dressing was 34%. With millet, 376 g./hectare of potassium iodide produced an increase of 28%. With sodium fluoride a maximum increase with barley was obtained using a dressing equivalent to 5 kg./hectare. Similarly, with millet a 40% increase was obtained with 940 g./hectare. G. W. Robinson.

Mitscherlich's method for determining the manurial requirements of soils. Gerlach (Z. Pflanz. Düng., 1926, **B5**, 489—505).—In considering the quantitative relationship between plant growth and growth factors, the increase in production of dry matter over the control should be used and not the actual production. It is further suggested that a given factor should be represented in the equation by the amount actually used by the plant. The effect constant for a given factor ("Wirkungswert") probably varies for different plants. In investigating the effect of any one factor, the others should be in favourable amount or intensity. The general applicability of Mitscherlich's equation is questioned. G. W. Robinson.

Application of the Neubauer seedling method to tropical soils. H. Vageler (Z. Pflanz. Düng., 1926, **B5**, 506—509).—The author gives data for the assimilability of potassium and phosphorus, respectively, in certain Colombian soils, using the Neubauer seedling method. The corresponding amounts soluble in 10% hydrochloric acid are also given. There is a rough correlation between potassium soluble in hydrochloric acid and potassium available to seedlings, the acid-soluble potassium being about twice or three times the amount taken up by seedlings. In the case of phosphorus the correlation is less obvious, and a much lower availability is indicated. The number of experiments is too small to suffice for any trustworthy conclusions. G. W. Robinson.

Influence of manuring etc. on the strength of straw of cereals. P. Stuch (Z. Pflanz. Düng., 1926, **A7**, 257—290).—Strength of straw in cereals is mainly influenced by the supply of nitrogen to the plant. A suitable nitrogen supply results in a strong development of the tissues. A superabundance of nitrogen causes a thinning of the cell wall and a general slackening in the exterior stratified cells; plants over-stimulated with nitrogen are prone to fungal diseases, which still further weaken the cellular structure. Phosphatic fertilisers invariably increase straw strength by thickening the cell walls and consolidating the external layers. The mechanical tissues develop earlier, and the straw is shown to withstand both increased tension and pressure. In some cases over-manuring with phosphate favours the incidence of fungal diseases, which may so weaken the cells as to outweigh the strengthening effect of the fertiliser. Potassic fertilisers both increase the strength

of straw and add to the plant's resistance to fungal attacks. A. G. POLLARD.

Relation between physiological acidity of fertiliser salts and soil acidity. H. KAPPEN and W. BERGEDER (Z. Pflanz. Düng., 1926, A7, 291—317).—Comparison is made of the effects of physiologically acid fertilisers on the "hydrolytic" and "exchange" acidity of soils (B., 1925, 140), their p_H values and buffer capacities (Jensen). The physiologically acid fertilisers increase both types of acidity, and decrease p_H values and buffer capacities. Ammonium sulphate is the most active in this respect; calcium superphosphate and potash salts do not increase acidity. The physiologically alkaline fertilisers—sodium nitrate, basic slag, and some potash salts—have the opposite effects. Slag is the most effective in this respect. Plant injury due to acidity produced by the use of acid fertilisers is not the result of the activity of free acids, but is due to enhanced "exchange" acidity. Soils exhibiting considerable "exchange" acidity may be treated with acid fertilisers without any appreciable increase in p_H values. For this reason lime-requirement determinations based on p_H measurements must fail. In soils of similar mechanical composition, values obtained for the neutralising power of soil for acids are comparable with the values for "hydrolytic" and "exchange" acidity. Comparison is not possible of soils with different physical composition. This conforms with the theory that the intensity of acidity is dependent on the quantity and state of disintegration of zeolitic silicates and humates in the soil. It is recommended, therefore, that determinations of the neutralising power of soil for acids should be supplemented by measurements of the "exchange" and "hydrolytic" acidity. A. G. POLLARD.

Nature and significance of the physiological soil reaction. B. DIRKS (Z. Pflanz. Düng., 1926, A7, 318—338).—It is shown that alkaline humates in soil can exert a definite alkaline reaction, and can neutralise free hydrogen ions up to p_H 6. Within the range p_H 6—7, in humus soils free from zeolitic material, plants may suffer from excessive alkalinity derived from the calcium humates. Calcium carbonate can be decomposed by humic acids within this range. In zeolitic soils no appreciable amount of calcium humates can exist, since the humate base can be withdrawn from solution into the insoluble zeolite complex. Only when there is an insufficiency of zeolitic material present does the plant suffer from alkali-toxicity. Calcium humates are decomposed by carbon dioxide within the range p_H 7—8·3. Under still more alkaline conditions begins the formation of "soda" soils. In these toxicity may be reduced owing to the formation of calcium carbonate by decomposition of the humate. Calcium humates are decomposed by boiling the soil extract for a short time, and the base may be directly titrated. Aqueous extracts are preferable to neutral salt extracts for this purpose, and give higher results, more closely paralleled by plant experiments. The optimum lime dressing for acid soils can be determined by an electrometric titration of a N-potassium chloride extract of the soil with lime water. The p_H value of the resulting liquid after boiling for 1 min. and filtering should not exceed 6·0 for zeolite-free soils. The end-point of the latter part of the experiment may, alternatively, be determined by titration with 0·01N-hydrochloric acid, using methyl-red as indicator. A. G. POLLARD.

Absorption of plant nutrients and formation of dry matter by varieties of millet under different manurial conditions. W. SCHLEUSENER (Z. Pflanz. Düng., 1926, A7, 137—165).—The course of absorption of plant nutrients by different varieties of millet during the growing period is similar to that of summer cereals, particularly barley. Production of dry matter lags behind the intake of plant nutrients, which is largely completed by the time of flowering. During the first three fortnightly periods of the growing season the course of absorption is not markedly influenced by the manuring. During the later periods the growth on manured plots was limited by drought. Only in the case of nitrogen was the course of absorption affected by manuring, and the greater the amount of nitrogen given the more did absorption precede formation of dry matter. The plant nutrient requirements of millet appear to be less than those of barley; nevertheless, this crop removes considerable amounts of plant food from the soil. On account of drought it was impossible to decide as to the response of millet to manuring. G. W. ROBINSON.

Translocation of potassium before and during the death of leaves of *Populus nigra* (black poplar) and *Hedera helix* (ivy) in autumn. T. SABALITSCHKA and A. WIESE (Z. Pflanz. Düng., 1926, A7, 166—173).—During the dying of the leaves of *Populus nigra* and *Hedera helix*, a 60—70% reduction of the potassium content occurs. The decrease is most marked shortly before and during coloration, and in the case of *Populus* begins in August. In the case of *Hedera*, leaves which remain green and do not fall show no decrease in potassium content. It is probable that a translocation of potassium to the permanent parts of these plants occurs in autumn. It is suggested that potassium may behave differently in other plants, and that the conclusions for *Populus* and *Hedera* may not be generally applicable.
G. W. ROBINSON.

Preparation and solubility of magnesium phosphates compared with those of calcium and aluminium, and their utilisation by oats and barley. E. UNGERER (Z. Pflanz. Düng., 1926, A7, 352—364).—The extremely variable constitution of laboratory preparations of magnesium phosphate is demonstrated. The phosphates of magnesium and calcium are all more soluble in carbon dioxide solutions than in water. The solubility of aluminium phosphate in water is decreased by the presence of carbon dioxide. Plant experiments indicate that the fertiliser value of these phosphates is of the same order as their increased solubility in the presence of carbon dioxide. A. G. POLLARD.

Manuring of fruit trees. I. II. T. WALLACE (J. Pomology, 1925, 4, 117—140; 5, 1—33).—A detailed series of nutritional experiments on apples, gooseberries, black currants, raspberries, and strawberries, extending over several years, is described. The plants were grown in sand cultures supplied with nutrient solutions. Deficiency of any one of the elements, nitrogen, potassium, phosphorus, calcium, magnesium, and sulphur, produced characteristic effects on the various plants,

influencing especially the type of foliage produced, the blossom formation, and the time of defoliation. Other data recorded relate to the effect of the treatments on the nature of the root systems, the amount of shoot growth, and the yield of fruit. The ratio of nitrogen to potassium and of potassium to magnesium in the food supply may be important. Some of the effects of deficiency of essential nutrients are so characteristic that they may prove useful for diagnostic purposes in the field. C. T. GIMINGHAM.

Determination of p_H by means of the quinhydrone electrode. T. ARND and W. SIEMENS (Z. Pflanz. Düng., 1926, A7, 191—204).—For the determination of the p_H of well buffered solutions, including soil extracts, commercial quinhydrone is as satisfactory as quinhydrone freshly prepared by the oxidation of quinol. In the absence of buffering substances, freshly prepared and recrystallised quinhydrone should be used. With soils having $p_H > 5 \cdot 0$, carbon dioxide should first be removed either by a current of air or, where the p_H is less than 8·5, a current of hydrogen.
G. W. ROBINSON.

Importance of hydrogen-ion concentration in the study of soils. G. WIEGNER and H. GESSNER (Kolloid-Z., 1926, **40**, 209—227).—The importance of hydrogen-ion concentration and methods of measurement in soils are discussed. Soils appear to be well buffered in general, their p_H value lying between 4 and 8. The chief sources of acidity are carbonic acid, humus, clay, and the hydrolysis of salts. Calcium carbonate and clay act as strong buffers towards acids, whilst humus acts as a strong buffer towards bases. Also, clay has a weak buffering effect towards bases, and similarly humus towards acids. Humus which is poor in lime has p_H 3·5—4·2, but the p_H is raised by increasing the lime content. The limits of p_H favourable to different types of plants are considered, and the effect of manuring is discussed. E. S. HEDGES.

Influence of water containing sulphuric acid on the germinating power of steeped grain. J. DEHNICKE (Z. Spiritusind., 1926, **49**, 336—337).—The prevention of mould during malting by the use of sulphuric acid is dependent upon both the concentration of the acid and the time it is in contact with the grain. After alternate steeping and aeration, the grain was soaked in water containing different amounts of sulphuric acid for varying periods, and the percentage of germinating grains compared with that obtained in a control experiment in which no sulphuric acid was used. Soaking for 30—60 min. in acid containing 0·049 g. of sulphuric acid/100 c.c. caused no diminution in the germinating power, but stronger acid caused a distinct falling off. F. R. ENNOS.

Nitrifying power of puzzuolana. II. C. SERONO and L. GUERCI (Annali Chim. Appl., 1926, **16**, 452—456; cf. B., 1925, 858).—When air is passed through a sodium hydroxide solution containing suspended puzzuolana, or when moist air is passed slowly over the earth, nitric acid is sometimes formed. Very few samples have this property, which is lost after a few days.
E. W. WIGNALL.

Acrid and poisonous qualities of seeds and cakes from *Cruciferæ*. G. JORGENSEN (Ann. Falsif., 1926, **19**, 454—459).—Two varieties of *Cruciferæ*, *Brassica juncea* and *Brassica niger*, yield the harmful allylthiocarbimide and the cake is first examined microscopically for seeds of these. If a qualitative test shows the presence of acrid mustard oil, determinations are made of its amount by formation of the thiosinamine and also of the percentage of nitrogen in the latter. When the nitrogen content of the thiosinamine exceeds 22% the material is harmful to cattle, even though it yields no more than 0·35% of mustard oil; when between 21% and 22% it is doubtful, but if less than 21%, and *Brassica juncea* is absent, no poisonous qualities are to be expected. When this value approaches 21% and *Brassica juncea* is present, it is advisable to try the effect of the cake on rats. F. R. ENNOS.

Action of potassium chloride on soils free from calcium. A. DEMOLON and E. NATIER (Compt. rend., 1926, **183**, 1121—1122).—The adsorption of potassium from potassium chloride by clay soils free from alkaline earths shows that even with soils showing a slightly acid reaction, sufficient calcium is retained adsorbed on the clay for an interchange of ions to take place (cf. Demolon, A., 1926, 678); and when this is the case the ratio (Ca displaced)/(K fixed) is approximately 0·5. When this ratio has a lower value the soil is deficient in calcium, and in this case an interchange of potassium and hydrogen ions occurs, so that after washing, the soil is less acid, the final p_H value being higher than the initial value. No change in the p_H is found when sufficient calcium is present. J. W. BAKER.

PATENTS.

Insecticidal composition and the like. H. B. GOODWIN, Assr. to THE LATIMER-GOODWIN CHEM. CO. (U.S.P. 1,604,774, 26.10.26. Appl., 6.5.24).—A small amount of casein is added to the insecticidal compound, which is mixed with water when required, a sufficient quantity of lime being included in the spray mixture to form calcium caseinate. H. ROYAL-DAWSON.

Manufacture of a manure. P. RIPPERT (E.P. 260,882, 26.5.26).—Peat, brown coal, or the like is made feebly alkaline in order to induce a conversion of the organic nitro-compounds into ammonia. The mass is then treated with oxygen, particularly nascent oxygen, *e.g.*, a 0·1% solution of potassium permanganate with addition of hydrogen peroxide. When the oxygen development is complete, cultures of aerobic yeasts which form protein in presence of ammonia are added.
E. H. SHARPLES.

Manufacture of phosphate fertilisers. E. L. LARISON, Assr. to ANACONDA COPPER MINING CO. (U.S.P. 1,604,359, 26.10.26. Appl., 24.3.26).—Ground phosphate rock, sulphuric acid, and phosphoric acid are mixed in such proportions that the thin mobile slurry thus formed will, on keeping without artificial drying, form a mass which is easily disintegrated into a granular powder suitable for use in fertiliser distributing machinery. E. H. SHARPLES.

XVII.—SUGARS; STARCHES; GUMS.

Chemical and physical de-liming of sugar solutions and syrups. H. A. C. VAN DER JAGT (Chem. Weekblad, 1926, **23**, 566—572).—Lime present in sugar juices is distinguished as "combined" when in the form

of salts other than the saccharate; this form is not precipitated by carbonatation, unless sodium or potassium hydroxide be present. The hydroxides may be added as such, or produced by the action of lime or alkali salts already present; sodium carbonate is to be preferred. The addition of lime and subsequent carbonatation causes no diminution of the combined lime content of artificially prepared sugar solutions, but produces a very marked diminution with cane juice; it is suggested that during flocculation of the colloidal particles, the non-sugars and especially calcium compounds are selectively adsorbed and carried down. The same explanation is suggested for the more complete purification of alkaline syrups by treatment with lime and carbon dioxide; if such syrups are first neutralised with acetic acid, de-liming is less complete. S. I. LEVY.

Influence of peptic digestion in the determination of total carbohydrates in cereal products. HARTMAN and HILLIG.—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Culture of the bacillus of retting of flax. V. OMÉLIANSKY and M. KONONOV (Compt. rend., 1926, **183**, 919—921).—Growth of the bacillus of retting of flax, *Granulobacter pectinovorum*, in presence of air, is favoured by addition of aerobic bacteria. A sterile suspension of flax fibres in water is inoculated with well retted material and heated for 15 min. at 115° (20 min. at 120° destroys the spores). It is then incubated at 32° in presence of *B. fluorescens liquefaciens*. Retting commences in 2—3 days, and is completed in a further 5—9 days. The same results are obtained with other aerobic bacteria, and also with pure cultures of *Granulobacter* under anaerobic conditions. H. E. F. NOTTON.

PATENT.

Manufacture of a manure (E.P. 260,882).—See XVI.

XIX.—FOODS.

Detection of sulphites in foodstuffs. A. E. PARKES (Analyst, 1926, **51**, 620—622).—10 g. of solid material mixed with about 10 c.c. of water in a 50—100 c.c. conical flask, or 10 c.c. of liquid are taken, 10 c.c. of dilute hydrochloric acid and 2 or 3 small pieces of marble are added, and the flask is closed by a rubber stopper carrying a thistle funnel of 3—4 mm. internal diam. bent twice, and having on each limb a bulb capable of holding about 2 c.c.; 2 or 3 drops of $0\cdot01N$-iodine solution and 1 drop of barium chloride solution are placed in the funnel as a seal through which all liberated gases must pass. When the action of acid on the marble has moderated the liquid is heated to boiling, and immediately the first drop of condensed liquid passes over into the funnel the colour of the iodine is discharged if sulphur dioxide is present, and a white opalescence due to barium sulphate is formed. Some fruit products contain certain volatile substances which discharge the colour of iodine, but do not give any reaction with barium chloride. It may be necessary to add an anti-frothing agent such as amyl alcohol. If the boiling be continued for 1—2 min. most of the sulphur dioxide will have been driven over (more $0\cdot1N$-iodine solution being added if necessary), and if the reagent and precipitate are washed out they may be matched against a known amount of sulphuric acid and barium chloride in a nephelometer and the process made partly quantitative. D. G. HEWER.

Variations in the composition of milk. J. F. TOCHER (Analyst, 1926, **51**, 606—613).—The percentage of total solids-not-fat of the bulked milk of one herd of cows from daily samples is much less variable (3 times less) than the percentage of total solids-not-fat in the bulked milk of different herds, so that a high percentage of butter fat is associated, in general, with a lower percentage than average of solids-not-fat. There is a high correlation between yield per milking and solids-not-fat per milking, and that between total butter fat per milking and yield per milking is also fairly high. Yield can be used as a measure of the total amount of solid matter in milk with a fair degree of accuracy. Although there is a correlation between solids-not-fat and butter fat percentages in the bulked milk of different herds, due to the high positive correlation between percentage of casein and of butter fat, the relationship is negative for the daily samples of bulked milk from one herd, owing to the small variation in the total solids from day to day. The percentage of casein in milk, lowest after calving, gradually rises to a maximum at the end of the lactation period. The correlation between butter fat and lactose is negative, accounting largely for the negative correlation between non-fatty solids (other than casein) and butter fat. Friesian cows were found to give a higher quantity of solids-not-fat in general, and of lactose in particular, per milking than Ayrshire or other cows, although Ayrshires showed higher percentages of butter fat and of solids-not-fat per milking. D. G. HEWER.

Resistance of fat-soluble vitamins to hydrogenation. L. RANDOIN and R. LECOQ (Ann. Falsif., 1926, **19**, 518—523).—Commercial cod liver oil (iodine value 134·39) was hydrogenated with reduced nickel catalyst at 180—190° for 6 hrs. to an iodine value of 28·69, and also at 120—130° for 8 hrs. to an iodine value of 64·93, and fed to rats which had been on a Sherman and Pappenheimer No. 84 diet and exhibited typical rachitic signs. A slow rate of growth was maintained, but in neither case were ophthalmic lesions inhibited, and the oil hydrogenated at the lower temperature was, if anything, more prejudicial to the symptoms than the harder one. Butter, particularly summer butter, was found to be less wanting in anti-rachitic properties than has been supposed, and margarines made with hydrogenated oils are inferior as sources of fat-soluble vitamins to those prepared with natural oils and fats.
D. G. HEWER.

Cellulose sausage casings. W. F. HENDERSON and H. E. DIETRICH (Ind. Eng. Chem., 1926, **18**, 1190—1194).—Details are given for the conversion, on a manufacturing scale, of high-grade purified cotton linters, by means of the viscose process, into thin-walled seamless cellulose tubes which are mechanically and chemically suitable for use as sausage casings. The new material has distinct advantages over the animal casings hitherto used. W. J. POWELL.

PATENTS.

Liquid-treating apparatus [for milk]. H. H. MILLER, Assr. to H. H. MILLER INDUSTRIES CO. (U.S.P.

1,603,970, 19.10.26. Appl., 26.10.22).—A vat for storing large quantities of milk without deterioration is fitted with a lining of German silver, surrounded by a heat insulating material, such as wood, and contains a series of pipes through which water at a constant temperature is circulated. Propellers are also provided for causing a gentle agitation of the milk in the vat. T. S. Wheeler.

Preparing flaked cereal food products. H. J. Heinz Co., Assees. of E. R. Harding (E.P. 245,474, 4.1.26. Conv., 3.1.25).—The grain is cooked with finely-divided pure cellulose and the mass dried incompletely, and then the original kernels are separated, dried, and flaked. E. H. Sharples.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Ethyl phthalate test. H. Wales (J. Assoc. Off. Agric. Chem., 1926, **9**, 476—477).—In testing for ethyl phthalate in drug products it is necessary first to remove derivatives of naphthalene, anthracene, and phenanthrene, as these may be oxidised to phthalic acid, and will consequently show a positive test with resorcinol. The precipitant used for the purpose is basic lead acetate, excess of which is removed by sodium carbonate. After extraction of the ester with light petroleum, hydrolysis, and conversion of the phthalic acid into anhydride, the latter is condensed with resorcinol. The fluorescence obtained if ethyl phthalate is present persists indefinitely without fading. If a positive test is obtained, it is advisable to confirm by distilling some of the original product and applying the test to the distillate. F. R. Ennos.

Analysis of Quinosol (2-hydroxyquinoline) preparations. R. Berg (Pharm. Ztg., 1926, **71**, 1542—1543).—The material is dissolved in water and titrated with a bromide–bromate solution, using indigo-carmine as indicator. The active quinoline compound is quantitatively brominated to 5 : 7-dibromohydroxyquinoline, any excess of bromine reacting with the colour indicator. As a check, a slight excess of the bromine solution may be added, then potassium iodide, and the liberated iodine determined by means of thiosulphate. S. I. Levy.

Determination of morphine in opium. E. C. M. J. Hollman (Pharm. Weekblad, 1926, **63**, 1337—1349, 1370—1380, 1393—1408).—The standard methods have been subjected to critical examination, and none is found satisfactory. The variation in opiums from different sources makes it impossible to recommend any one simple or composite method. The acidity of the opium varies, whilst the composition may be affected by ferments and oxidation; the solvent to be used for the extraction must be selected with due regard to the acidity; lime water is usually best, but the solution filters badly, and the crystals obtained are impure. Acid solutions extract the related alkaloids, and alcohol acts similarly. The p_H values and morphine contents of a large number of opiums from various sources show variations from 4·00 to 5·05 and from 5·99% to 15·00% respectively, the variations being entirely arbitrary; determinations by the methods of the Dutch and German pharmacopœias in the same samples gave differences in the morphine content up to 1·58%. The most favourable temperature for separation of the crystals is about 16°; lower temperatures considerably retard crystallisation. Related alkaloids and the usual impurities, besides adulterants and diluents, seriously affect both extraction and crystallisation; the influence of albumins on the separation is very unfavourable. Results are given of tests of the influence of time, agitation, etc. on the rate of separation. Trials on the most favourable p_H values for separation gave the best value as 8·84 for pure aqueous solutions. An apparatus for the determination of p_H values by means of the calomel electrode is described, also the results of a large number of examinations of opium from various sources tabulated with the morphine contents as determined by the Dutch and German methods; the results are discussed and some conclusions drawn, the most important being that the p_H determinations afford no help in deciding the best conditions for extraction and separation. The use of alcohol for extraction and crystallisation is discussed; it is found to promote contamination with lime, except where the procedure of the Japanese pharmacopœia is followed. The determination of the morphine finally obtained, whether this is weighed or titrated, may introduce errors; these are considered for the two methods. The use of adsorbent charcoal introduces considerable errors, which are less in acid than in alkaline solutions, but never negligible. The sources of error in the lime water and ammonia methods respectively are discussed in detail; the colorimetric, refractometric, and polarimetric methods are also considered. S. I. Levy.

New colour reaction for procaine [novocaine] and some other local anæsthetics, and its application to the determination of procaine. E. R. Riegel and J. F. Williams (J. Amer. Chem. Soc., 1926, **48**, 2871—2878).—A weighed sample, representing 10—15 mg. of procaine, in water (10 c.c.) is treated with 10% hydrochloric acid (0·5 c.c.), 2% sodium nitrite solution (1 c.c.), and concentrated ammonium hydroxide solution (1 c.c.), and, after 30 sec., the mixture is diluted to 100 c.c. 1, 1·25, 1·5, 2, and 2·5 c.c. of this solution, diluted to 50 c.c. in Nessler tubes, are compared with a standard tube, prepared by diluting a 0·1% solution of potassium dichromate (8·5 c.c.) to 50 c.c., the colour standard so obtained representing a total content of 0·25 mg. of procaine. No colour is obtained in the solution under test if the amount of procaine present in 10 c.c. is less than 0·05 mg. A concentration of 10—15 mg. in 10 c.c. gives the maximum colour; with concentrations below 10 mg./10 c.c., there is an absence of turbidity during the development of colour, indicating that the concentration is too low. The accuracy of the method is within 10%, provided that the temperature is maintained at 20° throughout. The reaction can also be used as a qualitative test for local anæsthetics, tutocaine, butyn, butesin, propaesin, benzocaine, and orthoform; saligenin gives a yellow colour with hydrochloric acid and sodium nitrite without the addition of aqueous ammonia. The method can also be applied to the determination of tutocaine. For the determination of mixtures of procaine and cocaine, synthetic mixtures must first be employed to establish a standard, since

cocaine, although it gives no colour in the test, removes some of the yellow substance from the solution when precipitated by the ammonia. Alternatively, the reaction is carried out in alcoholic solution, in which the cocaine does not interfere, but it is then necessary to establish colour values for procaine in alcohol.

F. G. WILLSON.

Critical inspection of the directions for the examination of essential oils and perfumes. W. TREFF (Z. angew. Chem., 1926, **39**, 1306—1309).—Xylol-musk exists in two modifications, a stable form, m.p. 112—113°, and a labile form, m.p. 105—106°. Formation of the stable form is influenced by inoculation of the melted musk or a concentrated alcoholic solution with a small particle of the stable form; also by crystallisation from ether, benzene, toluene, or light petroleum. The labile form exists only within narrow limits. Rearrangement is possible in the melted state, but the melt usually solidifies to crystals of the higher-melting modification. These facts explain the discrepancies in the recorded melting points of xylol-musk. The specified density of benzaldehyde (d^{15} 1·050—1·055), is considered to be too high. Chlorine-free benzaldehyde only oversteps the lower limit by reason of its benzoic acid content; a more suitable value is 1·049. The catalytic effect of small amounts of impurities and the sensitivity of many oils at high temperatures are shown in the distillation of pure linalool with the addition of small pieces of porous clay. Over 30% was converted into terpenes and resins. Small pieces of glass capillaries are much more suitable, and should always be used in the distillation of essential oils. Specified b.p. should define the velocity of distillation. In a series of distillations with a pure sample of cassia oil, different rates of distillation gave residues varying from 6—30%. A steam-distilled oil under similar conditions gave varying amounts of residue, and for this to be a minimum it is necessary to distil as rapidly as possible. In the saponification of acetylated alcohols an excess of alkali is essential for complete saponification and the prevention of the formation of ethyl acetate.

E. H. SHARPLES.

PATENTS.

Apparatus for producing acetic acid from acetaldehyde. E. G. THORIN, Assr. to STOCKHOLMS SUPERFOSFAT FAB. AKT. (U.S.P. 1,601,891, 5.10.26. Appl., 27.4.23. Conv., 19.9.21).—Acetaldehyde is oxidised in an upright tube filled with small pieces of solid material, the upper part of the tube being provided with a cooling jacket to condense the acetic acid and acetaldehyde, and the lower part with a heating jacket to drive off the acetaldehyde from the acetic acid. An acetic acid solution of the catalyst is admitted to the top of the tube, and between the two jackets there is an inlet for liquid acetaldehyde. At the bottom of the tube is an inlet for the gas containing oxygen and an outlet for the acetic acid formed. Waste gases are withdrawn from the top of the tube.

E. H. SHARPLES.

Manufacture of alkyl carbonates. J. A. S. HAMMOND, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,603,689, 19.10.26. Appl., 23.3.25).—Phosgene and an aliphatic alcohol are heated under reflux. The carbonate is recovered from the mixture by addition of an inert aliphatic solvent, which in the presence of water (which is added to form separate liquid layers) acts as a non-solvent for the alcohol. B. FULLMAN.

Manufacture of alkyl carbonates. H. G. MITCHELL, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,603,703, 19.10.26. Appl., 26.3.25).—Phosgene vapour is brought into contact with an aliphatic alcohol, the latter being in 100% excess. The mixture is allowed to remain until the product is formed. B. FULLMAN.

Making acetylsalicylic acid. D. A. NIGHTINGALE, Assr. to KETOID Co. (U.S.P. 1,604,472, 26.10.26. Appl., 22.7.24. Renewed 18.3.26).—Salicylic acid is treated with keten. B. FULLMAN.

Production of acylresorcinols. SHARP AND DOHME, Assees. of A. R. L. DOHME (E.P. 250,893, 6.7.25. Conv., 4.16.25).—Acyl resorcinols of the formula $C_6H_3(OH)_2\cdot COR$ ($R =$ alkyl), free from tarry by-products, are prepared by condensing fatty acids with about one-third of their weight of resorcinol, in the presence of zinc chloride (from less than half to less than one-fifth by weight of the fatty acid), at 125—145° (preferably 130—140°). The resorcinol is gradually added to the heated mixture of fatty acid and zinc chloride, heating continued for about 3 hours, and the product washed and distilled *in vacuo*. (Cf. A., 1926, 838.) B. FULLMAN.

Manufacture of solid polymers of formaldehyde. J. Y. JOHNSON. From I.-G. FARBENIND. A.-G. (E.P. 260,908, 26.6.25).—Water-soluble solid polymerides of formaldehyde, of any consistency from a grease to a dust, are obtained from gaseous mixtures of formaldehyde and water vapour (possibly also containing other gases and vapours) by removing the water vapour, preferably by rapid cooling, but also by other means. When the cooling method is used, the bulk of the water vapour and some formaldehyde condense and form a formaldehyde solution, which is removed, while the uncondensed formaldehyde passes on and is separated as a solid polymer. Examples are given of the catalytic oxidation of anhydrous and of 80% methyl alcohol, the cooling being effected in the two cases respectively by blowing the hot vapour obliquely on to the surface of strongly cooled 30% formaldehyde solution, and by passing the vapour from below into an inclined cooler.

B. FULLMAN.

Making cellulose esters of carboxylic acids. D. A. NIGHTINGALE, Assr. to KETOID Co. (U.S.P. 1,604,471, 26.10.26. Appl., 22.7.24).—Cellulose is treated with keten in the presence of a neutral liquid in which keten is soluble. B. FULLMAN.

Manufacture of alkaline-earth salts of the carboxylic acids of aromatic sulphonhaloalkali amides. "ESSEFF" CHEM. IND. & HANDELS A.-G. (E.P. 248,339, 30.1.26. Conv., 25.2.25).—The calcium salt of 4-calciumchlorosulphonamidobenzoic acid, which is obtained by the action of calcium hypochlorite on 4-sulphonamidobenzoic acid, is treated in aqueous solution with a sodium salt, whereby the calcium salt of 4-sodiumchlorosulphonamidobenzoic acid is formed.

A. DAVIDSON.

Manufacture of medicaments containing bismuth. CHEM.-PHARM. A.-G. BAD-HOMBURG, Assees. of A. LIEBRECHT (E.P. 237,912, 29.7.25. Conv., 29.7.24).—Products having high therapeutic qualities for the treatment of syphilis in both early and late stages are prepared by the action of one or more bismuth compounds on phosphatides such as lecithin. Reaction may be effected in the presence of organic solvents, and liquids suitable for injection are obtained by the addition of olive oil either before or after removal of the solvent. For example, 10 pts. of bismuth quinine iodide and 10 pts. of lecithin dissolved in ether are thoroughly mixed. On removal of the solvent an orange powder remains which is soluble in chloroform, carbon disulphide, and benzene. E. H. SHARPLES.

Methods of preparing a remedy for tuberculosis in men and animals. K. F. O. GRONSTEDT (E.P. 248,319, 1.9.25. Conv., 28.2.25).—Goats, dogs, or other animals are rendered immune to tuberculosis by injections of dead or living tubercle bacilli, or metal salts or the like, and killed by blood letting. The bone marrow is removed and, after drying at moderate heat, is prepared into tablets or powder permitting its use as a remedy. Extracts may be obtained by dissolving the finely ground marrow in water rendered faintly alkaline. E. H. SHARPLES.

Manufacture of anæsthetic and antiseptic compounds. E. RITSERT (E.P. 260,493, 15.4.26).—By interaction of esters of aminobenzoic acids, or their salts, and iodine- or bromine-substituted hydroxyaromatic or hydroxyheterocyclic sulphonic acids, or their salts, in the presence of solvents or blood serum, there are formed the corresponding difficultly-soluble sulphonates of the aminobenzoic esters, having anæsthetic and antiseptic properties. The following are described: the *di-iodophenol-p-sulphonate* of ethyl *p*-aminobenzoate, m.p. 225° (decomp.), and the corresponding *dibromo-p-sulphonate*, m.p. 193—200°; the 7-*iodo-8-hydroxyquinolinesulphonate* of propyl *p*-aminobenzoate, m.p. 225—228°; and the corresponding *di-iodophenol-p-sulphonate*, m.p. 224° (decomp.); and the *di-iodophenol-p-sulphonate* of *iso*butyl *p*-aminobenzoate, m.p. 222—224° (decomp.). B. FULLMAN.

Product for dissolving essential oils. E. G. THOMSSEN, Assr. to J. R. WATKINS Co. (U.S.P. 1,602,183, 5.10.26. Appl., 24.7.25).—The product consists of a flavouring extract of a flavouring material dissolved in the ester of a hydroxy-acid, or of a flavouring extract consisting of a flavouring material dissolved in ethyl lactate and containing no alcohol. B. FULLMAN.

Promoting catalytic reactions. C. R. DOWNS, Assr. to THE BARRETT CO. (U.S.P. 1,604,739, 26.10.26. Appl., 5.12.21).—An apparatus for carrying out catalytic reactions comprises a number of tubes containing the catalyst, surrounded by a liquid boiling under a pressure corresponding to the temperature it is desired to maintain. The refluxing portion of the liquid is employed to preheat the material passing to the catalyst. All overheating is avoided, even in carrying out strongly exothermic reactions, such as the oxidation of naphthalene by air to form phthalic anhydride. T. S. WHEELER.

Solvent recovery [from adsorbent carbon]. A. B. RAY, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,604,481, 26.10.26. Appl., 14.11.24).—The aqueous portion of the condensate, obtained by treating adsorbent carbon containing recovered solvent (*e.g.*, amyl acetate) with steam, is employed to cool the reactivated carbon, when the solvent dissolved in the aqueous liquid is adsorbed. The carbon is then employed in the usual way to recover fresh solvent. T. S. WHEELER.

Separating volatile chemicals [aniline from water]. W. E. LUMMUS (U.S.P. 1,603,851, 19.10.26. Appl., 27.8.20).—A mixture of aniline vapour and steam is passed into a fractionating column, from the bottom of which water free from aniline is withdrawn, and from the top a mixture of aniline (1 pt.) and steam (3 pts.). These vapours are partially condensed, and the solution of aniline in water, thus formed, is returned to the column. The remaining vapours are completely condensed, and run into a gravity separator, in which aniline and a solution of water in aniline are obtained. The solution is returned to the column. T. S. WHEELER.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Effects of dilution and stirring of a photographic developer. A. H. NIETZ and A. WHITAKER (Brit. J. Phot., 1926, **73**, 630—632, 645—647, 660—661, 676—677).—Time of development with a given exposure is not proportional to the dilution of the developer. The curve of density against logarithm of dilution is in general concave to the dilution axis. The following factors to which this may be due are considered in detail: (1) Penetration effects due to surface development (tanning) and to oxidation products in the emulsion layer; (2) penetration effects due to swelling; (3) effect of excess reducing agent; (4) aerial oxidation of the more dilute solutions; (5) possible variation of reducing energy with concentration; (6) hydrolysis; (7) effect of more vigorous stirring. All these points are tested experimentally, and it is considered that the effects of dilution are caused, in order of importance, by (1) the effect of hydrolysis in lowering the concentration of the active anion of the phenolate. No satisfactory explanation is suggested for the action of amines; (2) imperfect diffusion outwards of the bromide and other reaction products formed in the emulsion layer as development proceeds; proper stirring eliminates this effect and gives a differential increase of density, favouring dilute solutions; (3) aerial oxidation accounts for part of the falling off in density with the more dilute solutions; this only occurs with developers which oxidise rapidly.; (4) penetration effects caused by surface development and tanning not explained by (2). These conditions would affect only the concentrated solutions. W. CLARK.

Relation between time and intensity in photographic exposure. F. C. TOY (Brit. J. Phot., 1926, **73**, 704).—Emulsions were exposed to light at differing intensity levels, but for the same *It* value. With a certain low intensity level it was not possible to make more than

about 80% of the grains developable even with considerable increase in exposure. At higher intensity levels, however, it was very easy to get 100% changed. Using a slow, pure bromide emulsion, exposed to wave-length 3650 Å., the average number of development centres per grain increased rapidly with increasing exposure to a high intensity level, tending to infinity at a certain exposure. With corresponding exposure at a lower intensity, the average number of centres per grain tended to a low maximum with increasing exposure. The results fit in with recent observations of Jones and Hall (B., 1926, 1030). W. CLARK.

Panchromatisation of photographic plates for use in the ordinary spectrum. E. VITERBI (Gazzetta, 1926, **56**, 612—620).—Of a number of different compounds and mixtures of compounds tried with respect to their ability to render ordinary photographic plates panchromatic, solutions having as basis a mixture of pinachrome and pinachrome-violet gave the best results. The effect is improved by addition of ethylcyanine; and a bath containing 220 c.c. of water and 2 c.c. each of 1 : 1000 solutions of pinachrome, pinachrome-violet, and ethylcyanine is recommended. The plate is immersed in this bath for 3—4 min., washed for 1—2 min. in flowing water, and dried in an ordinary draining rack. If the 220 c.c. of water are replaced by 150 c.c. of water and 70 c.c. of 95% alcohol the washing may be omitted. T. H. POPE.

PATENTS.

Photographic medium. M. C. BEEBE and H. V. HERLINGER, Assrs. to WADSWORTH WATCH CASE Co. (U.S.P. 1,604,674, 26.10.26. Appl., 22.4.25).—A stable light-sensitive varnish is produced by introducing a free halogen into a varnish consisting of hydrophobic colloids, metal salts capable of combining with free halogen, and a solvent, the salts being present to the point approaching saturation. The proportion of free halogen is such as to combine with a substantial portion of the salts and to avoid the presence of free halogen after the metallic halides have formed. W. CLARK.

Reducing the intensity of photographic silver images. K. E. TAESCHNER (E.P. 260,892, 7.6.26).—Silver images are reduced by immersion in a preliminary bath containing an alkali chromate or dichromate and potassium ferricyanide, followed by a second bath in acid sodium thiosulphate. The first bath may contain sugar. W. CLARK.

Photographic [photomechanical] processes. H. WADE. From THE WADSWORTH WATCH CASE Co. (E.P. 260,460, 4.12.26).—A photographic medium consists of a phenol condensation resin with a halogen source as sensitiser. Suitable sensitisers are halogen-liberating compounds such as halogen-substituted hydrocarbons, *e.g.*, iodoform, bromoform, magenta (hydrochloride), or rhodamine alone or in combination with iodine, metal alkyls with a slight excess of iodine, or metallic halides. Hydrogen iodide or any compound liberating a halogen under the action of light is available. As solvent for the resin it is preferable to use one which will give a mobile varnish-like medium. Development is by immersion in a solvent such as alcohol and water. W. CLARK.

Utilisation of biochemical light-sensitising extracts in the art of photographic materials. S. E. SHEPPARD, Assr. to EASTMAN KODAK Co. (U.S.P. 1,602,590, 12.10.26. Appl. 21.1.26).—See E.P. 235,210; B., 1925, 694.

Photographic light-sensitive material containing [A] tellurium, [B] selenium. S. E. SHEPPARD, Assr. to EASTMAN KODAK Co. (U.S.P. 1,602,591—2, 12.10.26. Appl., 6.6.24).—See E.P. 235,211; B., 1925, 694.

XXII.—EXPLOSIVES; MATCHES.

Properties of initiating explosives. H. KAST (Z. ges. Schiess- u. Sprengstoffw., 1926, **21**, 188—192).—Tests carried out in 1909 showed that the effect of mercury fulminate increased when it was compressed. The initiating effect was measured by modifying Esop's test by mixing crystalline T.N.T. with increasing amounts of paraffin and noting the amount of paraffin necessary to prevent any considerable compression (1 mm.) of a lead cylinder by the initiation of the phlegmatised T.N.T. by the initiator under test. It was found that detonators containing a layer of fulminate compressed at 1500 kg./sq. cm. had a greater initiating effect than those with fulminate compressed at 600 kg./sq.cm. Similar results were obtained with lead azide. No experimental support was found for statements that the greater effect of aluminium detonators was due to a thermo-chemical action of the metal. Increased power obtained by adding potassium chlorate to mercury fulminate is not due to increased brisance, as the brisance of the mixture remains practically the same as that of the fulminate alone. The favourable effect must be attributed to an increase in the ease of ignition and quicker establishment of the detonation wave. Storage of technical mercury fulminate in atmospheres of 50%, 80%, and 100% relative humidity showed that it is not hygroscopic, but in admixture with commonly occurring impurities such as mercurous chloride or in presence of potassium chlorate or gum arabic it shows considerable hygroscopicity. The hygroscopicity of fulminate in presence of potassium chlorate, which is itself also non-hygroscopic, is due to reduction of chlorate by impurities present in technical fulminate. The sublimate formed when cyanuric triazide is heated is found to be unchanged cyanaric triazide. Several errata in a paper by H. Kast and A. Haid are corrected (see B., 1925, 153). S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Testing of disinfectants by the Rideal–Walker method. Q. MOORE, JUN. (J.S.C.I., 1926, **45**, 472—474 T).—The standardisation of the broth was found to be an extremely important factor in the test. Variation in the method of neutralising the broth to phenolphthalein, by titration in the cold or at the b.p., altered the final reaction of the broth and resulted in differences in the phenol coefficient values of disinfectants. An increase of, approximately, one-third in the phenol coefficient values of disinfectants was obtained when tests were made using p_H 7·6 broth as compared with p_H 6·9 broth.

The p_H 7·6 broth gave results for disinfectants comparable with the presently accepted Rideal–Walker values.

Coal-tar disinfectants. A. C. Tait (J.C.S.I., 1926, **45**, 415—417 t).—A brief review. The method of Chick and Martin for the determination of the bactericidal coefficient is recommended.

Potassium permanganate absorbtion, "chlorine number" and chlorination of water. W. Olszewski (Z. angew. Chem., 1926, **39**, 1309—1310).—Determinations of the "chlorine numbers" and permanganate absorbtions of a series of artificially contaminated waters confirm the results of Froboese and Keiser (cf. B., 1926, 222). For the examination of drinking waters the importance is emphasised of the ratio of the permanganate absorbtion to the chlorine number in conjunction with the bacteriological examination for the detection of contamination by protein degradation products. For the control examination of swimming-bath waters the permanganate absorption and chlorine number should both be determined. E. H. Sharples.

Automatic hydrogen-ion control of boiler feed water. H. C. Parker and W. N. Greer (J. Amer. Water Works Assoc., 1926, **16**, 602—616).—A description is given of the apparatus used for electrometrically determining and automatically recording the p_H of feed water after treatment with caustic alkali, and for automatically controlling the supply of chemical required to bring the feed water to the desired p_H. With automatic hydrogen-ion control the average alkalinity of boilers may be lowered with safety, and more uniform control is obtained of the alkalinity of individual boilers and of the average alkalinity of boilers as a whole. It is cheap, and requires little attention; is the only method which automatically compensates for condenser leakage, adds chemicals automatically in proportion to the flow, and in such proportion as to compensate approximately for the acidity or salt content of the feed water, and provides a method for the prevention of corrosion in the preheating sections and the feed lines. W. T. Lockett.

Determination of colon bacterium. C. J. Lauter (J. Amer. Water Works Assoc., 1926, **16**, 625—630).—A review of research work carried out at Washington, D.C. Filtration Plant laboratory on brilliant-green–bile medium. Results indicate that the presence of *B. coli* in raw and filtered water can be detected by the bile medium with a high degree of accuracy, and in a shorter time than that required for the performance of the lactose presumptive and confirmatory tests. As a confirmatory medium, in place of eosin–methylene-blue plates, the brilliant-green–bile medium has given good results. (Cf. B., 1926, 302.) W. T. Lockett.

Diagnostic value of neutral-red–lactose–peptone media for the *coli–aerogenes* group. F. R. Georgia and R. Morales (J. Amer. Water Works Assoc., 1926, **16**, 631—641).—The neutral-red–lactose–peptone medium proposed by Stokes and developed by Chamot and Sherwood as a presumptive medium for the *coli–aerogenes* group would appear to be better than any medium so far proposed, and results indicate that when a typical contrast reaction is obtained further confirmation is unnecessary. No differentiation of *coli* and *aerogenes* sections of the group can, however, be effected by the medium. In tests on samples from a variety of sources, 96·8% of tubes showing a typical reaction were confirmed as containing members of the *coli–aerogenes* group. Of all the tubes confirmed, 76·5% gave typical reactions, 18·1% were completely reduced, and 5·4% showed no reduction. From tubes completely reduced, in addition to a member of the *coli–aerogenes* group, other organisms capable of completely reducing neutral-red were isolated, and these when present prevent the typical contrast reaction being obtained. Absence of any reduction may be due to attenuated forms or to the presence of certain of the more unusual members of the *coli–aerogenes* group that do not have the ability to reduce neutral-red, or which do so very slowly. The medium has no inhibitory action. W. T. Lockett.

Alkalimetric determination of the hardness of industrial waters. C. Belcot (Bull. Soc. chim., 1926, [iv], **39**, 1648—1652).—The values obtained for the permanent hardness of a water not containing alkali carbonates or hydrogen carbonates by the method of Treadwell or of Pfeifer (Z. angew. Chem., 1902, **15**, 9, 198) often disagree with those obtained by difference. The discrepancy is due to the partial solubility of magnesium carbonate, which influences the determination of the temporary hardness. Hence, in a water containing alkaline-earth and alkali hydrogen carbonates, magnesium sulphate and chloride, and sodium carbonate, the total carbonate and hydrogen carbonate is determined by titration. The total hardness is then determined, and the permanent hardness determined after boiling the water and neutralising the alkali hydrogen carbonates with hydrochloric acid. S. K. Tweedy.

Patents.

Dewatering, compressing, and drying industrial wastes and sewage solids. F. W. Manning (U.S.P. 1,604,652, 26.10.26. Appl., 17.6.25).—The filtered solids are built up upon a spiral filter wall, and are moved along the wall. W. G. Carey.

Water softener. H. A. Kern (U.S.P. 1,604,124, 26.10.26. Appl., 24.4.24).—A permanent liquid product containing sodium aluminate and caustic soda. H. Royal-Dawson.

Process for clarifying water. H. A. Kern (U.S.P. 1,604,125, 26.10.26. Appl., 26.5.24).—Calcium hydroxide mixed with a solution of sodium aluminate and caustic soda is added to water. H. Royal-Dawson.

Solidified water-softening compound. H. A. Kern (U.S.P. 1,604,126, 26.10.26. Appl., 24.9.24).—A sufficient amount of soda ash is added to a solution of sodium aluminate to take up the solvent as water of crystallisation. H. Royal-Dawson.

Removing gases from boiler feed water by a vacuum (E.P. 254,707).—See I.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

FEBRUARY 4, 1927.

I.—GENERAL; PLANT; MACHINERY.

Removal of precipitates from settling vessels without running off the supernatant solution. Steen (Chem.-Ztg., 1926, **50**, 961—962).—Considerable economy is often obtained if sediments can be removed from large volumes of liquid without pumping the whole bulk of liquid. A cylindrical settling tank with conical bottom may effect this, especially if to the bottom sludge outlet a pressure vessel is connected to balance the liquor head. Several conical sumps so fitted up may be connected to one large tank. If a uniform mixture of coarse and fine sediment is desired the settling vessel may take the form of a ring narrow in section at the bottom. The turbid liquid is fed in centrally and overflows the outer edge of the ring-shaped vessel into a gutter, from which it is removed. The coarse precipitate is deposited at the bottom, and the finer as the liquid ascends the outer wall, but all grades become uniformly mixed in the channel at the bottom, and may be pumped as a thick slurry into a final settling vessel placed centrally within the ring. C. Irwin.

Automatic apparatus for the determination of water. R. Kattwinkel (Chem.-Ztg., 1926, **50**, 927).—An apparatus for the determination of water in a substance by heating with benzene, toluene, or other organic liquid consists of a distillation flask fitted with a reflux condenser having a ground glass joint in the neck of the flask. The issuing vapours enter the condenser through a side tube at the lower end, and the condensed liquid runs into a measuring burette, which consists of an extension of the lower end of the inside tube of the condenser through the outer walls, and terminates in a graduated portion to which a stopcock is attached. The water collects in the burette, and the lighter organic liquid overflows back into the flask through the tube by which it entered the condenser.
A. R. Powell.

Apparatus for the determination of moisture by distillation with xylene. J. Pritzker and R. Jungkunz (Chem.-Ztg., 1926, **50**, 962—963).—The accurate determination of moisture by continuous distillation with xylene requires attention to the form of the cooler. That used by the authors is of the internally-suspended type with outer vapour jacket. 15 c.c. of xylene are used, the weight of substance under test being adjusted as suitable. A graduated tube is connected to the bottom of the cooler with a connexion to enable clear water-free xylene to be returned to the distillation flask. The tube containing the turbid mixture is then centrifuged. A uniform correction of $+0\cdot1$ c.c. is applied per 5 g. of water. This method is suitable for the determination of moisture in butter, sugar, peat, soap, and the like, and also for such inorganic compounds as sodium chloride. Ferrous and copper sulphates retain 1 mol. of water of crystallisation when distilled with xylene. C. Irwin.

Lubricants for ground glass joints. Bradley and Wilson.—See VIII.

Patents.

Furnace. I. Hechenbleikner and T. C. Oliver, Assrs. to Chemical Construction Co. (U.S.P. 1,603,462, 19.10.26. Appl., 25.3.21).—The nozzle of a burner is directed into a combustion chamber provided with an annular baffle surrounding the burner and a predetermined zone of the burner flame. A ring of gas inlets is arranged around the baffle, and these are constructed and arranged for imparting a whirling motion of the gas about the zone of the flame beyond the baffle.
H. Holmes.

Heat exchangers. Griscom-Russell Co., Assees. of J. Price (E.P. 248,712, 4.1.26. Conv., 3.3.25).—In a tubular heat exchanger where one liquid flows through two (or more) bundles of tubes in series, all the tubes at one end are supported in one tube plate, but at the other end the tube plate is subdivided, and one section is connected to another by means having a certain amount of "give," comprising, substantially, four elbows and a straight pipe. B. M. Venables.

Pulverising apparatus. C. J. Reed, Assr. to J. G. Berryhill (U.S.P. 1,603,639, 19.10.26. Appl., 1.12.21).—One end of a horizontal high-speed rotary drum is provided with a stationary closure, and rollers, interconnected by a frame, move freely within the drum to co-operate with the surface for grinding. The frame carries means for continuously removing material from the drum, and the upper end of the stationary closure carries means for removing the fine dust produced.
H. Holmes.

Grinding members for ball, drum, and tubular mills. O. Kordt (E.P. 261,664, 4.8.26).—A grinding member, preferably of steel, is of approximately equal diameter vertically and transversely, and is formed with flattened and plane surfaces at varying angles. Suitable shapes are described. H. Holmes.

Grinding, pulverising, or disintegrating mills. W. A. Cloud (E.P. 262,033, 20.7.25).—A disintegrator of the type comprising several rings of intercalating pins, or of similar construction, is provided with a tangential outlet for the ground material, which can be moved round the casing to the most advantageous position. B. M. Venables.

Retort. I. B. Newbery (E.P. 261,575, 19.1.26).—The retort has an outer vertical and cylindrical casing, within which is a series of superposed rotating shelves. The material passes through the retort by being scraped

progressively from one shelf to the next below. Around the bottom of the retort is a series of upwardly-directed steam jets, whilst a steam-jet ejector is fitted in the off-take pipe at the top of the retort. The gases and vapours are thus removed while a partial vacuum is maintained in the retort. The shelves are progressively heated from below. R. A. A. Taylor.

Drum dryer. D. J. Van Marle, Assr. to Buffalo Foundry & Machine Co., Inc. (U.S.P. 1,603,886, 19.10.26. Appl., 17.1.25).—Liquid to be dried is delivered from a supply tank below a rotary drum to a feeding pan provided with an inclined bottom extending tangentially to the drum surface above the tank, with its inner edge spaced from the surface. The liquid is thus fed to the drum in a direction opposite to the rotation, and the excess, not adhering to the surface, passes back to the tank over the spaced edge. H. Holmes.

Dryer. T. H. Rhoads, Assr. to Proctor & Schwartz, Inc. (U.S.P. 1,604,074, 19.10.26. Appl., 19.6.26).—The drier is divided into two sections side by side, each provided with a fan for circulating air. The articles to be dried are carried on trucks within a tunnel extending through both sections. The air circulates first through one section, then through the other, and back through the first section, and means are provided for diverting a proportion of the air through the tunnel in direct contact with the articles. H. Holmes.

Dryer. W. M. Schwartz, Assr. to Proctor & Schwartz, Inc. (U.S.P. 1,604,079, 19.10.26. Appl., 5.12.23).—The casing is made in sections, each comprising inner and outer metallic plates separated by non-conducting material. The edges of the sections are offset to provide overlap joints between the sections. The edges of the plates are disconnected at the offsets, and non-conducting strips are placed between the sections to prevent transmission of heat from the inner to the outer plates. H. Holmes.

Centrifugal separators. A. W. Empson (E.P. 260,720, 15.9.25).—A centrifugal separator is provided with a number of baffles forming ducts, parallel to the wall and to the axis of the bowl, through which the fluid under treatment has to flow. The fluid may be constrained to flow through several ducts in series or series–parallel. B. M. Venables.

Mixing machinery or apparatus. J. B. Parnall and W. W. Veitch (E.P. 260,745, 10.10.25).—An open pan is rotated, and into it, off centre, so as to touch or nearly touch the side at one point, depends a stirring device free to rotate under the influence of the material in the pan. The stirring device is counterbalanced so that it may be easily lifted out, and, if desired, a fixed scraper may be provided in the pan at a point opposite the rotatable stirrer. B. M. Venables.

Mixing methods and apparatus [for liming sugar juices etc.]. Gilchrist & Co., Assees. of R. W. Shafor (E.P. 247,541, 25.1.26. Conv., 13.2.25).—The ingredients of the mixture are circulated together in a closed cycle, and baffles are provided for dividing the stream and producing sudden changes in the direction of flow of the subdivisions. Successive groups of the baffles may be staggered for re-distributing the mixture between adjacent subdivisions. Means are provided for regulating the temperature of the stream. H. Holmes.

Apparatus for making emulsions. L. Kirschbraun (U.S.P. 1,603,546, 19.10.26. Appl., 9.4.24).—A horizontal open-ended conduit is mounted within a mixing chamber provided with inlets for the materials to be mixed, and agitating elements are provided in the conduit and in the chamber. H. Holmes.

Distilling apparatus for volatile liquids. H. A. Hills (U.S.P. 1,604,353, 26.10.26. Appl., 14.1.21).—A still for purifying cleansing liquids has a nozzle placed horizontally and adjacent to the bottom of the still, which injects steam into the liquid. The nozzle has a sleeve which forms an annular chamber, and this is supplied with fluid from the bottom of the still simultaneously with the exhaust from the nozzle, the injected steam and fluid being deflected upward. W. G. Carey.

Removing volatile fluids from solids containing same. R. E. Wilson, Assr. to Baltimore Gas Engineering Corp. (U.S.P. 1,603,568, 19.10.26. Appl., 1.6.22).—The solid material and an absorbent are subjected to relative movement while maintaining them in close proximity. H. Holmes.

Apparatus for separating solids [coal] from liquids. Woodall–Duckham (1920), Ltd., and R. Krall (E.P. 261,850—1, 28.8.25).—The apparatus described in E.P. 219,651 (B., 1925, 271), which is especially suitable for drying coal, is provided with (a) a stationary or revolving hood to collect the water sprayed out of the material, and (b), a special form of joint between the rotating perforated screen and the stationary lower part of the apparatus to prevent re-mingling of coal and water. B. M. Venables.

Centrifugal separation of particles suspended in a fluid. Tirage & Ventilation Mécaniques (E.P. 261,617, 10.5.26. Conv., 28.11.25).—The fluid passes through the separating chamber between axially-alined inlet and outlet passages. The inlet passage is provided with stationary vanes for imparting gyratory, centrifugal motion to the fluid, and the outlet passage with stationary vanes, oppositely disposed to those in the inlet passage, for restoring to the fluid the pressure lost within the chamber. The two sets of vanes may be mounted one at each end of a drum, the chamber being constituted by an annular space between the drum and an outer casing. This space may be provided with an annular partition dividing the chamber into inner and outer zones communicating freely at each end. H. Holmes.

Filtering device. J. J. Naugle (E.P. 261,204, 14.12.25).—A number of filter leaves are supported on a framework or yoke within a pressure-tight casing. The yoke and leaves can be rotated by means of trunnions extending through the casing, and the interior of each leaf communicates with the outside through a trunnion, and the duct is provided with a sight glass. In operation the prefilt, comprising, say, a sugar solution with a filter aid such as "suchar" (activated carbon) in suspension, is circulated through the casing from top to bottom, and the filtrate is drawn off through the majority (but not all) of the leaves until the casing is practically full of suchar and residue. The circulating

feed is then shut off and the pulp exhausted as dry as possible by means of the hitherto unused leaves. During the filtering any leaf that passes muddy liquor may be shut off and, at the end of the cycle, changed for a sound one. After filtering, the residue is subjected to a hot reverse wash, the heat being maintained by live steam injected into the casing, and after washing the suchar and residue are sluiced out by jets of water (or other liquid) within the casing, but outside the leaves. The filters are rotated slowly during filtration, but rapidly during washing and discharging.

B. M. Venables.

[Continuous] pressure filters. J. A. McCaskell (E.P. 261,218, 18.1.26).—A number of filter discs are slowly rotated in a closed tank partly filled with the prefilt under pressure. Each leaf comprises a number of segments, the interiors of which are put, at the proper time, by means of conduits in the shaft and bearings of the filter, in communication with either a filtrate drain or a supply of air under a higher pressure than the filtering pressure. The supply of the pressure air is pulsated by a valve and shakes off the cakes of residue when the segments are raised above the liquid. The dislodged residue is caught by deflectors and discharged from the casing by means such as a worm conveyor.

B. M. Venables.

Apparatus for crystallisation. E. C. R. Marks. From Grasselli Chemical Co. (E.P. 261,085, 11.8.25).—A body of saturated solution is maintained in circulation, passing in the upward direction through the crystalliser proper, which is a vessel larger at the top than at the bottom, the average increase of section being less than a 60° cone. A multitude of small crystals are maintained in suspension in the crystalliser, and are caused to grow by the addition of supersaturated solution through the side of the crystalliser. Large crystals drop to the bottom of the crystalliser, and are removed by a conduit separate from and below the inlet for circulating solution. To prevent clogging of the return pipes etc. the solution is slightly heated when passing through the upper part of the crystalliser, and it may be reconditioned, *e.g.*, by evaporation, before re-use. B. M. Venables.

Purifying steam, vapours, and gases centrifugally. W. Alexander (E.P. 261,642, 17.6.26).—The steam etc. enters the separating chamber through convergent, parallel flow openings in the end plate separated by oblique or curved vanes which impart a vortex motion to the steam. The outer walls of the openings are parts of a concentric circular extension of the chamber wall. The end of the chamber adjacent to the drain outlet is sloped inwards, and the diameter of the chamber may be constant or may increase towards the drain outlet end. The admission end plate may be flat or convex towards the chamber. The steam outlet may be at the same end as the steam inlet, and its entrance may be provided with a double lip to prevent entraining of liquid with the outgoing steam. H. Holmes.

Controlling the temperature of heated air in drying and like operations [for crops etc.]. B. J. Owen (E.P. 261,446, 23.4.26).—A louvred or other shutter or damper on the outlet duct of the air-heating apparatus is controlled by a bimetallic strip thermostat mounted in the duct. The strip comprises two blades of metals, such as brass and mild steel, having widely different coefficients of expansion, and is curved to extend adjacent to the curved wall of the duct. The strip is fixed to the wall at one end, and carries at the other end a lug extending through a slit in the wall for connexion to the shutter through bell-cranks and connecting rods. H. Holmes.

Producing cooling liquids. Stratmann & Werner, and F. Werner (E.P. 260,760, 24.10.25).—A cooling mixture of low freezing point, which does not set free hydrochloric acid or other residues which corrode metal containers, is made by adding, under pressure, a solution of crystallised calcium chloride, which has previously been slowly heated, to a solution of magnesium chloride kept in motion at a high velocity. W. G. Carey.

Liquefaction apparatus. J. L. Schlitt and W. Dennis, Assrs. to Air Reduction Co., Inc. (U.S.P. 1,604,240, 26.10.26. Appl., 5.2.26).—A mixture of gases to be separated is introduced into tubes extending through a vaporiser section of a liquefaction column. Means are provided for withdrawing a gaseous product from the tubes, and means within the tubes retard the backward return of liquid formed therein in contact with the gaseous mixture. H. Holmes.

Apparatus for liquefaction and separation of gaseous mixtures. C. C. Van Nuys, Assr. to Air Reduction Co., Inc. (U.S.P. 1,604,248—9, 26.10.26. Appl., 6.12.24; cf. B., 1925, 575, 791).—[A] The gaseous mixture is delivered to a chamber beneath a liquid-containing compartment within the rectifying column, and tubes passing through the compartment connect the chamber to a header above the compartment. Means are provided for delivering liquid from the chamber to a rectifier above the compartment, and for effecting preliminary rectification of the liquid formed in the tubes by contact with the incoming mixture. [B] The mixture is subjected to selective liquefaction to form a liquid containing the constituents and a residual gas, which is withdrawn and expanded, and the expanded gas is used to cool the entering mixture. The liquid is delivered to the top of a rectifier, and means are provided for withdrawing from the top of the rectifier an effluent having substantially the composition of the entering gaseous mixture, and for withdrawing separately the vapour from the rectified liquid. H. Holmes.

Antifreezing mixture. G. A. Schultheiss (U.S.P. 1,605,377, 2.11.26. Appl., 25.9.25).—This consists of an oil dissolved in an alcohol and a calcium chloride solution. H. Royal-Dawson.

Separation of the constituents of ternary gaseous mixtures. C. C. Van Nuys, Assr. to Air Reduction Co., Inc. (U.S.P. 1,607,323, 16.11.26. Appl., 11.7.25).—See E.P. 255,104; B., 1926, 1000.

Separation of gases liquefying at different temperatures. G. Claude, Assr. to Lazote, Inc. (U.S.P. 1,605,646, 2.11.26. Appl., 30.1.20. Conv., 10.2.14).—See F.P. 475,346; B., 1916, 31.

Crucible. J. D. M. Smith, Assr. to W. & J. George, Ltd. (U.S.P. 1,609,096, 30.11.26. Appl., 8.6.26. Conv., 5.6.25).—See E.P. 252,901; B., 1926, 650.

Oil-fired furnace. C. Hawkins (E.P. 261,126, 27.8.25).

Dry packing insulation against heat exchange with protective mantle. F. Bohle and R. Schröder (E.P. 252,198, 17.5.26. Conv., 16.5.25).

Means for regulating the admission of steam to a heat accumulator. Siemens-Schuckertwerke Ges.m.b.H. (E.P. 255,491, 17.7.26. Conv., 18.7.25).

Regulation of liquid level in a pressure vessel. G. Campbell (E.P. 245,445, 28.12.25. Conv., 5.1.25).

Containers for storage and transport of gases. Union Générale Co-operative (E.P. 252,386, 20.5.26. Conv., 20.5.25).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Industrial conversion of coal into organic products of technical use. G. Patart (Chim. et Ind., 1926, **16**, 713—729).—The direct treatment of coal, *e.g.*, distillation, hydrogenation, etc., offers only a partial solution to the problem of its conversion into pure organic substances of use industrially. Some technical points are discussed relating to the conversion of coal into such products, *e.g.*, alcohols, acids, ketones, etc., by the direct combination of carbon monoxide and hydrogen under pressure in the presence of a catalyst (cf. B., 1925, 83). As this reaction proceeds, the concentration of the inert gases in the residual gas mixture increases to a point at which further reaction becomes practically ineffective, when the apparatus is purged and refilled. The loss of the active gases carried away with the inert gases on purging makes it necessary to use a process for gasifying the coal giving a minimum concentration of inert constituents. It is preferable to carbonise bituminous coals at high or at low temperatures, or possibly under pressure, and subsequently to gasify the coke, rather than to gasify directly; the synthesis of alcohols etc. is thus most favourably and economically carried out in association with a gas works. Technical applications of the products of the process are briefly discussed.

A. B. Manning.

Oxidation of pyrites as a factor in spontaneous combustion of coal. S. H. Li and S. W. Parr (Ind. Eng. Chem., 1926, **18**, 1299—1304).—The rate of oxidation between 25° and 100° of pyrites in coal in general increases with the fineness of division and with the temperature. At the ordinary temperature oxidation is favoured by high moisture content of the coal and by the presence of a current of oxygen, possibly because the moisture as it is removed is replaced by oxygen in a highly reactive state. In presence of certain types of clay, which probably contain sulphur-oxidising bacteria, oxidation may be as rapid at 25° as it is at 100° when the clay is absent. This is of importance, because it seems that in favourable circumstances the rate of oxidation of the coal itself may become serious above about 80°. Marcasite oxidises at about the same rate as pyrites does, but the process is facilitated by the ease with which it disintegrates into fine particles. R. Cuthill.

Rôle of metallic colloids in suppression of detonation. H. L. Olin, C. D. Read, and A. W. Goos (Ind. Eng. Chem., 1926, **18**, 1316—1318).—The efficiencies of various substances as anti-knock materials have been compared by adding them to the petrol used to drive an engine and observing the minimum load necessary to produce knocking. The efficacy of a solution of lead tetraethyl which has been heated under pressure so as to produce some colloidal lead is not impaired if the latter is removed with an activated carbon, and seems, by comparison with an experiment in which undecomposed lead tetraethyl was used, to be due solely to the undecomposed compound. A colloidal solution of nickel has no measurable anti-knock effect. It thus seems that free metallic particles suspended in the combustion zone of the cylinder do not necessarily suppress detonation (cf. Charch, Mack, and Boord, B., 1926, 570). R. Cuthill.

Apparatus for the determination of moisture [in peat] by distillation with xylene. Pritzker and Jungkunz.—See I.

Patents.

Fuel briquettes. G. Plochmann (U.S.P. 1,600,065, 14.9.26. Appl., 7.7.25. Conv., 2.5.25).—Lignites which have retained their woody structure are subjected to preliminary drying, so regulated that the material does not become actively hygroscopic. The pores of the dried material are charged with bitumen by exposure to the distillation gases of some suitable carbonaceous material, and the product is briquetted. S. Pexton.

Production of briquetting material. W. Pruijs, Assee. of H. Schrader (E.P. 250,925, 6.4.26. Conv., 15.4.25).—Coal dust is fed on to a rotating plate, whence it is thrown off and falls in the form of a hollow cylindrical cascade. A spray is arranged in the axis of, and below, the plate and rotates counter to the plate. It projects the binding material against the inner wall of the cascade of coal. R. A. A. Taylor.

Obtaining hydrogenation gas for hydrogenating coal and hydrocarbons from the waste gases of the hydrogenation. F. Bergius (E.P. 244,730, 16.11.25. Conv., 18.12.24).—The purified gaseous products from the hydrogenation of coal are treated with steam at a high temperature (about 1100°), whereby the hydrocarbon gases are converted into carbon monoxide and hydrogen is generated. The mixture of these two gases is then treated with steam at a lower temperature (300—500°), when, in the presence of a suitable catalyst, the carbon monoxide reacts to give carbon dioxide and hydrogen. The carbon dioxide is removed by washing.

R. A. A. Taylor.

Fuels from coal and oil mixtures. W. E. Trent (E.P. 261,594, 15.2.26).—Finely pulverised coal is treated with oil and then heated, when the oils distilled from the coal are recovered for the treatment of a further quantity of coal. R. A. A. Taylor.

Production of carbon. F. Fischer and H. Tropsch (E.P. 252,152, 5.5.26. Conv., 15.5.25).—Carbon monoxide, or a gas containing it, having been freed from sulphur compounds, is passed at 500°, preferably under pressure, over a catalyst of finely divided iron free from sulphur. Carbon is deposited on the iron and carbon dioxide is formed. The process is continued until the separated carbon includes not more than 10% of iron.

R. A. A. Taylor.

Production of lamp black. H. A. Pagenkopf (U.S.P. 1,606,380, 9.11.26. Appl., 16.9.24).—Oil and old tyres are burned in a restricted supply of air. The carbon is recovered from the smoke, first by deposition on a cooled revolving surface, then by passage through cooled and restricted spaces, and finally by spraying with water. R. A. A. Taylor.

[Peat] carbonising apparatus. W. C. E. Domnick, Assr. to Deutsche Verkohlungs- & Destillationsges. m.b.H. (U.S.P. 1,604,738, 26.10.26. Appl., 14.7.24. Conv., 29.12.23).—An apparatus for the distillation of peat comprises 12 retorts arranged in a ring. The process employed is similar to that described by W. Dominick (cf. B., 1925, 59). T. S. Wheeler.

Apparatus for the destructive distillation of organic matter. W. K. Ljungdahl, Assr. to A. W. Cooper (U.S.P. 1,603,343 and 1,603,345, 19.10.26. Appl., [A] 25.1.23; [B] 19.1.26).—[A] Wood is placed in a chamber divided into separate compartments which are heated by gases from a furnace. One of the compartments forms a part of the closed circulatory system for the heating gases. The furnace is intermittently cut off from the circulatory system, when air is admitted to the former, and the heated products from it are used to heat another compartment. A cooling compartment is provided for the charge. [B] Each of a series of furnaces is connected to a regenerator and to a common equalising chamber. Any regenerator can be disconnected from the equalising chamber, but furnace gases may be caused to heat the disconnected regenerator. The gases from the equalising chamber pass to a retort. R. A. A. Taylor.

Apparatus for distillation. W. K. Ljungdahl (U.S.P. 1,603,344, 19.10.26. Appl., 13.6.23).—The retort (for wood) is heated by steam radiators. A system of pipes is connected to the retort, and is provided with means for cooling and condensing the vapours flowing through it, after which it discharges into the atmosphere. The separated condensates are collected in a still which is heated by exhaust steam from the radiators. R. A. A. Taylor.

Kiln for destructive distillation of organic matter. W. K. Ljungdahl, Assr. to A. W. Cooper (U.S.P. 1,603,346, 19.10.26. Appl., 19.1.26).—A long retort has an inlet door at one end and an outlet door at the other. At the opposite ends also are ports, one for admitting and one for exhausting gas, but the port at the inlet end is at least one charge-carrier length from the door. R. A. A. Taylor.

Tunnel kiln. W. K. Ljungdahl, Assr. to A. W. Cooper (U.S.P. 1,603,347, 19.10.26. Appl., 19.1.26).—In a kiln which has a heating and a cooling compartment which are to be traversed successively by the charge, means are provided for circulating a cooling gas, for cooling the gas, and, if required, for introducing to the cooling compartment gas from the heating compartment. R. A. A. Taylor.

Oil-shale retort. C. E. Barnhart (U.S.P. 1,604,253, 26.10.26. Appl., 14.2.25).—The retort consists of a number of horizontal roasting hearths, above and below each of which is a heating flue. The shale is drawn over the topmost hearth by a rotating screw, and falls on to the second hearth, and so on, thus passing across each hearth until discharged from the retort. Revolving gas-tight partitions at various points between certain hearths divide the retort into separate heat-treating sections. W. N. Hoyte.

Treating bituminiferous materials. J. J. Jakowsky (U.S.P. 1,602,819, 12.10.26. Appl., 16.5.22).—The apparatus comprises a tubular retort, in which is a stationary heat-transmitting element, spaced from the walls of the retort, and having a flat face in contact with the material, which is fed into the retort and swept across the face of the heat element. Bases and vapours of distillation are withdrawn by a conduit. H. Moore.

Electrical treatment [cracking] of gases and vapours [hydrocarbons]. H. R. Rowland, Assr. to C. and C. Developing Co. (U.S.P. 1,601,771, 5.10.26. Appl., 15.11.23).—An apparatus for subjecting hydrocarbons to the action of the silent electric discharge comprises a cylindrical chamber, which contains a wire electrode, connected to insulators at the top and bottom. The vapours under treatment pass up through the cylinder, and are subjected to the action of a discharge passing from the wire to the walls of the vessel. To prevent deposit of conducting material, *e.g.*, carbon, on the insulators between the wire and the chamber a portion of the material under treatment is sprayed in liquid form upon them. T. S. Wheeler.

Removal of deposited material from cracking plant. L. C. Huff, Assr. to Universal Oil Products Co. (U.S.P. 1,603,541, 19.10.26. Appl., 26.1.23. Renewed 27.12.24).—An expansive fluid, which is a non-supporter of combustion, is injected into the body of the deposited carbonaceous material, thereby disrupting it, and facilitating its removal. W. N. Hoyte.

Reflux column. W. W. Holland, Assr. to Standard Oil Co. (U.S.P. 1,603,772, 19.10.26. Appl., 12.5.24).—A reflux column for attachment to a (petroleum) still is fitted with a number of horizontal plates, spaced about 1 ft. apart, and each perforated with about 70 $\frac{1}{4}$ in. holes per square foot. From each hole depends a tube about 5 in. in length, and these tubes form the only method of passage from one space to the next. T. S. Wheeler.

Distillation [of petroleum oil]. W. F. Faragher, W. A. Gruse, and F. H. Garner, Assrs. to Gulf Refining Co. (U.S.P. 1,601,729, 5.10.26. Appl., 25.5.21).—Petroleum oil or the like is fractionally distilled by heating it in a still below the cracking temperature. The oil is caused to circulate vertically in the body of the oil and laterally at the top and bottom, by means of currents of gas, to promote mixing and uniformity of temperature, the distilled vapours being collected. H. Moore.

Treating water-in-oil emulsions. Breaking petroleum emulsions. M. De Groote, Assr. to W. S. Barnickel & Co. (U.S.P. 1,606,698—9, 9.11.26. Appl., [A] 23.12.24; [B] 29.4.26).—(A) An emulsion of water in oil is treated with an insoluble substance, which is wetted by water, can be dispersed colloidally in it, and is free from a fatty acid radical, *e.g.*, magnesium resinate or calcium cresylate. The emulsion is then passed through a bed of fine sand, or other substance composed

of insoluble particles, separated by capillary spaces, and capable of being wetted by oil, when separation rapidly ensues. (B) The emulsion is passed through a bed of fine sand resting on an earthed plate, between which and a disc placed above the sand a potential of about 11,000 volts is maintained. T. S. Wheeler.

Process of treating hydrocarbons. H. I. Lea (U.S.P. 1,606,246, 9.11.26. Appl., 26.5.22).—Hydrocarbons and chlorine are passed at an elevated temperature over alumina, whereby aluminium chloride is formed. The excess hydrocarbons are then acted upon by the aluminium chloride. R. A. A. Taylor.

Treatment of carbonaceous material. H. Nielsen, Assr. to B. Laing (U.S.P. 1,605,761, 2.11.26. Appl., 7.3.25).—See E.P. 198,385; B., 1923, 760.

Drying of finely-divided carbonised fuel. W. Broadbridge, E. Edser, and W. W. Stenning, Assrs. to Minerals Separation North American Corp. (U.S.P. 1,606,867, 16.11.26. Appl., 20.11.22. Conv., 29.11.21).—See E.P. 193,947; B., 1923, 438 A.

Process for purifying oils and the like. H. M'Ridge and W. R. Hodgkinson (U.S.P. 1,608,339, 23.11.26. Appl., 3.3.23. Conv., 4.3.22).—See E.P. 203,354; B., 1923, 1118.

Vibratory screens or sieves [for coal etc.]. T. Wood (E.P. 261,561, 18.12.25).

[Apparatus for] manufacture of gas retorts or other pipes from clay or the like. R. Gay and Gibbons (Dudley), Ltd. (E.P. 261,512, 24.9.25).

Apparatus for use in the discharging of retorts. H. J. Toogood, and R. Dempster & Sons, Ltd. (E.P. 261,059, 9.7.25).

Gaseous fuel burners. C. F. Hammond and W. Shackleton (E.P. 261,808, 2.6.25).

Apparatus for separating solids from liquids (E.P. 261,850—1).—See I.

Retort [for treating shale] (E.P. 261,575).—See I.

Producing ammonium sulphate [from coal gas] (E.P. 262,320).—See VII.

Purifying and concentrating sulphur dioxide (U.S.P. 1,606,299).—See VII.

Solvents (E.P. 259,293).—See XX.

III.—TAR AND TAR PRODUCTS.

Tar from a commercial low-temperature retort. H. A. Curtis and H. A. Beekhuis (Chem. Met. Eng., 1926, **33**, 666—669).—Some properties of a tar produced by the carbonisation of a West Virginia coal in a "Carbocoal" retort at 600—650° are described. The m.p. and the free carbon in the pitch are linear functions of the volume per cent. of the tar distilled. The tar was repeatedly fractionated, cuts being taken at 10—15° intervals up to 270°, and the tar acids were separated from each fraction. The sp. gr. of the tar acid fractions pass through a minimum at b.p. 224—238°. Other samples of the tar acids were methylated and the methyl ethers fractionated; the sp. gr. again pass through a minimum, the curve of average equivalent weights showing a corresponding break. The condensation products of the tar acids and formaldehyde were inferior to those obtained by using pure phenol. The condensation products with sulphur chloride may serve as acid-resisting varnishes. The neutral oils were fractionated; if care is taken to remove completely the sulphuric acid used in extracting the bases, the lower-boiling fractions of the neutral oil retain their light yellow colour for several months, whilst the fractions above 220° darken slowly. All the neutral oil fractions contain sulphur (0·4—0·6%), which is not readily removed. The neutral oils are highly unsaturated, absorbing, for example, up to 50% of their weight of chlorine; they are, however, not so readily attacked by potassium permanganate. Liquid-phase cracking of the neutral oils is very slow below 400°, whilst above 450° coke formation becomes rapid. The effect of cracking any fraction is to widen the boiling range, but the same relative difference in the boiling range between fractions is maintained even after extensive cracking.

A. B. Manning.

Behaviour of lignite producer tar and lignite low-temperature tar when heated under pressure. A. Erdély (Brennstoff-Chem., 1926, **7**, 359—361).—Producer tar oils and low-temperature tar oils, obtained from Tata lignite, give a lower yield of cracked oils than American gas oil, the optimum temperature in each case being 450°. When heated with hydrogen under pressure the lignite tar oils give 23—30% of cracked oils, as against 33% for the gas oil. Tar oils that have been refined with sulphuric acid and sodium hydroxide give the same yield as the gas oil, but the refining losses are heavy. The benzine obtained from the gas oil does not darken when kept, whereas that from the tar oils does. The presence of hydrogen under pressure increases the benzine yield and lessens the losses due to the formation of coke and gas. W. T. K. Braunholtz.

Patents.

Process for obtaining pressed asphalt compositions and similar products (E.P. 248,752).—See IX.

Drying of finely-divided carbonised fuel (U.S.P. 1,606,867).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Patents.

Manufacture of vat dyestuffs of the anthraquinone series. A. G. Bloxam. From Chem. Fabr. Griesheim-Elektron (E.P. 261,510, 23.9.25).—Arylketodihydro-*p*-thiazines, dihalogenated in the methylene group (cf. G.P. 379,318, A., 1924, i, 1118), are condensed with aryl-*o*-diamines or *N*-alkyl or -aryl derivatives of these. The products are vat dyes, if at least one reaction component is an anthraquinone derivative. Use of primary *o*-diamines yields dyes which are not fast to alkali, but which can be converted into alkali-fast dyes by alkylation. *E.g.*, 7·5 pts. of anthraquinonedichloroketodihydro-1 : 2-thiazine, 4·8 pts. of 1 : 2-diaminoanthraquinone, 80 pts. (by vol.) of nitrobenzene, and 3·5 pts. of anhydrous sodium acetate are heated for 2—3 hrs. at 140—150°. The yellowish-brown product obtained by distilling off the nitrobenzene in steam, after purification, dyes cotton yellow from a blue vat. It is ethylated by heating 4 pts. of it with 30 pts. of

ethyl toluenesulphonate at 200° for 7 hrs. The melt is diluted with alcohol and the yellow dye filtered off, washed, and purified by hypochlorite solution. The structure of the condensation products has not been determined. A. Davidson.

Azo dyestuffs. W. Carpmael. From I.-G. Farbenind. A.-G. (E.P. 261,568, 1.1.26).—Four azo *dyes* of the class described in E.P. 245,865 (B., 1926, 233), but not specifically mentioned therein, are prepared by coupling diazotised 4-amino-4′-hydroxydiphenylsulphone-2 : 3′-dicarboxylic acid with 2-naphthol-8-sulphonic acid, and by coupling diazotised 2-amino-4-sulpho-4′-hydroxydiphenylsulphone-3′-carboxylic acid with methyl ketol, with 2-amino-8-naphthol-6-sulphonic acid, and with *o*-amino-*p*-tolyl ether, the product in the last case being diazotised and coupled with salicylic acid. A. Davidson.

Dry diazo composition. F. Keller and K. Schnitzspahn, Assrs. to Grasselli Dyestuff Corp. U.S.P. 1,607,462, 16.11.26. Appl., 20.1.26. Conv., 30.1.25; cf. E.P. 21,227/94; B., 1895, 962).—Organic diazo compounds, when mixed with about an equal weight of crystalline alum or aluminium sulphate, about two-thirds dehydrated, can be stored for long periods without deterioration. T. S. Wheeler.

Preparation useful for the production of dyestuffs. A. Zitscher, Assr. to I.-G. Farbenind. A.-G. (U.S.P. 1,608,284, 23.11.26. Appl., 2.6.24. Conv., 11.6.23).—See E.P. 217,594; B., 1925, 541.

Process for making azo-dyestuff from barbituric acid. G. de Montmollin, F. Straub, and J. Spieler, Assrs. to Soc. Chem. Ind. in Basle (U.S.P. 1,606,209, 9.11.26. Appl., 1.12.25. Conv., 24.12.24).—See E.P. 257,797; B., 1926, 910.

Sulphur dye and process of making same. W. Merte, Assr. to Kalle & Co. A.-G. (U.S.P. 1,606,254, 9.11.26. Appl., 17.10.24. Conv., 12.8.22).—See F.P. 588,874; B., 1926, 149.

Manufacture of a new thioindoxyl derivative. Soc. Chem. Ind. in Basle (E.P. 246,156, 14.1.26. Conv., 14.1.25).—See Swiss P. 111,997; B., 1926, 943.

Manufacture of ice colours (E.P. 261,542).—See VI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Decomposition of raw vegetable fibrous material by chlorine. H. Wenzl (Papierfabr., 1926, 24, 809—817).—Comparisons are made between the actions of chlorine gas, chlorine water, and hypochlorite solution upon both pine-wood meal and straw under standard conditions. With wood, the highest percentage of chlorine used and the lowest ratio of combined chlorine to hydrochloric acid produced are shown when the chlorination is carried out in carbon tetrachloride, hydrolysis under these conditions being negligible. The values are a minimum and maximum, respectively, when chlorine water is used, hydrolysis of the chlorine then being complete. With straw, chlorine gas and hypochlorite are similar in action, whilst chlorine water differs in producing a smaller increase of copper number and decrease of baryta number. Further comparison is made by the effect of these three reagents upon the pentosan and lignin content and upon the yield of pure cellulose. Differences in the nature of the reactions with chlorine water and gas are ascribed to the fact that in the former case a substitution action predominates, whilst in the latter oxidation is the more important. Acid produced in the chlorine water process exerts a strongly hydrolysing action on the cellulose, resulting in a smaller yield of pure cellulose. Decomposition of wood by a single chlorination is successful only if a vigorous preliminary treatment with alkali is given. On a price basis, the chlorine gas method is the most economical. B. P. Ridge.

Viscosity of cuprammonium solutions of cotton cellulose. F. C. Hahn and H. Bradshaw (Ind. Eng. Chem., 1926, **18**, 1259—1260).—Viscosity measurements by the falling ball method show that linters yield more viscous solutions than does long-staple cotton. If η is the viscosity of a solution containing m g. of solute/100 c.c., and η' is the viscosity of the solvent, then $\log \eta/\eta'$ is a linear function of m, except at the higher concentrations (cf. Farrow and Neale, B., 1924, 506). R. Cuthill.

Viscose. III. Conditions of xanthation. M. Numa (Cellulose Ind. Tokyo, 1926, **2**, 321—335).—The quantity of carbon disulphide required to produce complete xanthation is 40—75%; the highest yield of xanthate and the most stable conditions correspond with about 50%. When the temperature of ripening is comparatively high it has little influence on the quantity of combined alkali, but the combined sulphur decreases rapidly. Hence it is contended that the combined alkali and combined sulphur do not necessarily vary proportionally. The most favourable temperature of xanthation is 15°. During the ripening the changes in the colloidal properties of the viscose gel particles and in the structure of the viscose sol take place at different velocities. The optimum duration of ripening is between 1 and 4 days, corresponding with the optimum properties of the filament or film. When this duration is surpassed the combined alkali and sulphur decrease, and the viscose tends to become unstable. The optimum degree of ripening may be recognised by the appearance of the precipitate formed on decomposing viscose of a certain concentration with 20% sulphuric acid. At the optimum point the regenerated cellulose is in the form of long fibrous particles, and should not be short or powdery. The dispersity of the viscose gel increases with the advance of ripening up to a maximum on the third day, and then decreases. J. F. Briggs.

[Detection of abnormal treatment of] artificial silk (cellulose hydrate). O. Faust and K. Littmann (Cellulosechem., 1926, 7, 166—168).—Determinations such as that of the copper number, breaking strain, etc. do not give reliable indications of the treatment to which a sample of cellulose has been subjected. The following method, due to Weltzien (B., 1926, 737), is shown to yield useful information concerning the previous history of the sample. The dry material is treated with water, then with 4% sodium hydroxide, then with water again, and is then dried, the percentage change in the fibre-length at each stage being determined, and the procedure is then repeated on the same sample. A normal fibre increases to 104% of its original length on treatment

with water, shrinks to 103% with 4% sodium hydroxide solution, increases again to 104% with water, and on drying shrinks to slightly less than its original length. On repetition of the treatment closely similar results are obtained. With a fibre which had previously undergone abnormal mechanical strain by being loaded with a weight of 50 g., the fibre length after water treatment was 100·5%, after alkali treatment 98·5%, after second water treatment 99·2%, on drying 95·5% of the original. When the process was repeated this material gave results similar to those of a normal fibre. The effect of abnormal mechanical strain, which may result in a change in the dyeing properties and lustre of the fabric, can thus be corrected by alkaline immersion. A fibre which had undergone abnormal chemical treatment, viz., immersion in 0·48% sulphuric acid solution and dehydration at 60°, gave the following results. After water treatment the fibre length was 101·5% of the original, after alkali treatment 100·5%, after second water treatment 102%, on drying 94%. A repetition of the procedure gave 106·5%, 106%, 107%, and 100%. The effect of acid cannot, therefore, be counteracted by alkaline immersion, as the treatment results in a fibre which shows abnormally high changes in length with water and sodium hydroxide. This may be due to the formation with acid of alkali-soluble products which are removed during the first treatment, and the fibre is thus rendered more penetrable. Treatment of the fibre with dilute hydrochloric acid has a similar effect, but acetic acid causes little change. Excessive bleaching results in abnormally high changes in length when the fibre is treated with water and alkali in the manner described above (106%, 105%, 107%, and 97%), and still more abnormal results when the process is repeated (108·5%, 108%, 109%, and 98·5%). It is therefore claimed that the method not only detects fibres which have undergone severe treatment, but also gives an indication of the conditions to which the sample has been subjected.

W. J. POWELL.

Cellulose benzoate. K. ATSUKI and K. SHIMOYAMA (Cellulose Ind. Tokyo, 1926, **2**, 336—345).—Cellulose dibenzoate is prepared by treating regenerated or normal cellulose with 35% sodium hydroxide solution, ageing for 24 hrs., and shaking with benzoyl chloride dissolved in benzene. The proportions used are $C_6H_{10}O_5 : 4NaOH : 10C_7H_5OCl$. The reaction is finished off by heating at 50—60° for 1—2 hrs. The dibenzoate from regenerated cellulose gives clear solutions in chloroform and acetone of very low viscosity, that from normal cellulose is not completely soluble, and the viscosity is very high. The films have a tensile strength of 5 kg./sq. mm. and elongation of 5%; they are very brittle.

J. F. BRIGGS.

Celloluse lacquer solvents etc. VAN HOEK.—See XIII.

PATENTS.

Washing, cleaning, or otherwise treating wools, silk, soiled fabrics, or other fibrous textile materials. E. C. DUHAMEL, and COMP. GÉN. DES INDUSTRIES TEXTILES (E.P. 259,969, 20.5.25. Conv., 20.5.24).—The material to be washed or cleaned is first treated with hot, relatively concentrated, suint liquor for a considerable time before entering the first of a series of washing bowls containing concentrated suint liquor. Apparatus for this purpose comprises an arrangement of a vertical shaft, down which the material passes while being impregnated with liquor, conveying lattices, beater rolls for opening and distributing the material uniformly in thin layers, squeeze rollers to express mud and grease, and a series of washing bowls in which the suint liquor is maintained in a successively cleaner condition.

B. P. RIDGE.

Cleaning wool, fabrics, etc. B. PAYNE (U.S.P. 1,608,418, 23.11.26. Appl., 21.6.24).—Bentonite is agitated with water, the mixture kept for maximum hydration, and the supernatant colloidal solution used for washing textile materials.

A. J. HALL.

Improving cellulosic materials, in particular vegetable textile fabrics. L. DE WOLF (E.P. 252,360, 12.5.26. Conv., 25.5.25).—The rate of defibreing, scouring, and bleaching of cellulosic materials by the action of sodium hydroxide or alkaline phenolate solutions is greatly enhanced by use of a mixture of the two, the former being in excess. With certain fibres a bath containing at least 5% of sodium hydroxide and 1% of phenol results in scouring, lanification, mercerisation, or even gelatinisation and bleaching in one operation. The efficiency of oxidising agents such as permanganates or alkali hypochlorites, when added to such a bath, is increased by the action of the phenol in solution.

B. P. RIDGE.

Making artificial silk and staple fibre. M. HÖLKEN (U.S.P. 1,607,400, 16.11.26. Appl., 26.8.21. Conv., 2.12.18).—A precipitating bath is prepared by mechanically softening a hard water, to deprive it of its temporary hardness, and adding a substance which destroys the permanent hardness, but does not affect the product precipitated in the bath.

H. ROYAL-DAWSON.

Treating artificial silk fibres. OBERRHEINISCHE HANDELSGES.M.B.H. (E.P. 260,935, 19.1.26. Conv., 6.11.25).—The spinning of artificial silk is facilitated by improving the adhering and intertwining properties of the fibres. For this purpose a roughening treatment is given prior to spinning or carding, in which substances such as talc, cement, oxides, or salts are fixed upon the fibres by precipitation, drying, or by adhesives. Curling or crisping, and pounding, pressing, fulling, or centrifuging the fibres in addition to the above treatment, further improve their spinning qualities.

B. P. RIDGE.

Manufacture of a rubberised fibre composition. W. G. O'BRIEN, Assr. to THE GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,606,595, 9.11.26. Appl., 31.7.22. Renewed 12.1.26).—The fibres are first separated, rubber particles caused to adhere to them, the product is treated with a liquid comprising a mixture of rubber precipitant and rubber solvent, and finally cured. The solvent is removed from a rubber solution by successive treatments with liquid mixtures containing increasing proportions of alcohol from 23 to 60%.

B. P. RIDGE.

Manufacture of cellulose-containing solutions. A. CLASSEN (E.P. 261,494, 3.9.25. Addn. to 236,281; B., 1926, 707).—The preparation of cellulose solutions in concentrated hydrochloric acid is improved by the use of catalysts in the form of metal oxides upon which the action of the acid is negligible, or nearly so. Increased

efficiency is obtained by employing two or more catalysts (such as metals and metal oxides in close contact) and by increasing the active surface. Thus the catalyst may be in the form of fine wire, or as metal wool, the wires being either superficially oxidised or provided with active surface layers, or it may be deposited on metallic or non-metallic carriers which are themselves either active or inert. B. P. Ridge.

Stabilised cellulose ester. H. S. Mork, Assr. to Lustron Co. Inc. (U.S.P. 1,607,474, 16.11.26. Appl., 12.3.21).—Cellulose acetate, and the artificial silk prepared from it, are rendered more stable to the action of heat, carbonisation being inhibited, by incorporation, before or during the manufacturing process, of 0·5—2% of the potassium or sodium salt of oleic, salicylic, or other organic acid. T. S. Wheeler.

Paper making. J. J. Gannon, L. W. Maille, and F. B. Wells, Assrs. to The Ohio Boxboard Co. (U.S.P. 1,606,162, 9.11.26. Appl., 6.4.25).—A layer of filler material is superimposed upon another of pergameneous material, the surplus water being afterwards expressed from the combined layers in a direction away from the pergameneous material and through the filler. B. P. Ridge.

Making waterproof products. L. Kirschbraun (U.S.P. 1,606,427, 9.11.26. Appl., 16.1.25).—Waste fibrous products, containing a bituminous waterproofing binder with a dispersing agent, are beaten up with an aqueous vehicle at a temperature near to the melting point of the binder, thus causing separation of the fibres. The bituminous material is precipitated on the fibres to render them non-sticky, and the stock is felted on a paper machine. B. P. Ridge.

Process of paper making. C. Ellis, Assr. to Ellis-Foster Co. (U.S.P. 1,607,517, 16.11.26. Appl., 8.4.26).—A hot aqueous wax dispersion is added to a hydrocellulose pulp. H. Royal-Dawson.

Treating paper pulp. C. Ellis, Assr. to Ellis-Foster Co. (U.S.P. 1,607,518, 16.11.26. Appl., 12.4.26).—The hydrogen-ion concentration of the pulp is adjusted so as to increase the absorption of a wax emulsion. H. Royal-Dawson.

Paper product. C. Ellis, Assr. to Ellis-Foster Co. (U.S.P. 1,607,519, 16.11.26. Appl., 21.4.26).—The product consists of a rosin-sized paper of hydrocellulose containing wax in excess of rosin. H. Royal-Dawson.

Production of transparent and waterproof paper. A. E. Maze, Assr. to Ellis-Foster Co. (U.S.P. 1,607,552, 16.11.26. Appl., 30.12.25).—An emulsion of paraffin wax is incorporated in the fibre resulting when moist paper pulp is subjected to a beating operation. H. Royal-Dawson.

Manufacture of products containing cellulose. C. Dreyfus (E.P. 262,157, 6.6.25).—See U.S.P. 1,546,211; B., 1925, 707.

Hank yarn dryers. H. Haas (E.P. 261,686, 25.9.26. Conv., 9.3.26).

Method of cocoon-storage by cooling and drying. K. Kobori, and Kanegafuchi Boseki Kabushiki Kwaisha (E.P. 261,093, 13.8.25).

[Apparatus for] manufacture of lustrous fabrics. F. Wolf, Assee. of H. Kuhn (E.P. 259,940, 28.9.26. Conv., 13.10.25).

Spindles of spinning machines for artificial silk and the like. Harbens (Viscose Silk Manufacturers) Ltd., W. E. Sharples, and General Electric Co., Ltd. (E.P. 261,499, 16.9.25).

[Cellulose] lacquer for shoe heels etc. (U.S.P. 1,607,516).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Patents.

Discharge effects on [textile] materials containing cellulose acetate. Brit. Dyestuffs Corp., Ltd., and L. Smith (E.P. 262,254, 25.11.25).—Clear white and coloured discharge effects on cellulose acetate silk dyed with those dyes commonly used for this type of silk are obtained in the usual manner by means of reducing agents containing sodium formaldehyde-sulphoxylate, provided that thiocyanates such as calcium, barium, and ammonium thiocyanate are present. A satisfactory discharge paste contains 15 g. of formosul (sodium formaldehyde-sulphoxylate), 70 g. of 3% gum tragacanth solution, and 10 g. of a solution of calcium thiocyanate (*d* 1·44). A. J. Hall.

Treatment of ["immunised"] cotton fibres, preparatory to dyeing. P. Karrer (E.P. 249,842, 11.3.26).—Cotton partially esterified with the sulphonic acids of benzene, toluene, or naphthalene (cf. E.P. 195,619; B., 1924, 128), and heated with ammonia, aliphatic or aromatic primary, secondary, or tertiary amines, or with hydrazine or hydrazine derivatives, in suitable solvents, acquires an affinity for acid dyes such as tartrazine, Orange II, and sulphorhodamine; *e.g.*, 10 pts. of cotton partially esterified by means of *p*-toluenesulphochloride are heated for 6 hrs. with 50 pts. of a 25% aqueous solution of ammonia in an autoclave at 100°. A. J. Hall.

Dyeing process [for wool]. Badische Anilin- u. Soda-Fabr. (E.P. 242,233, 8.8.25. Conv., 30.10.24).—Sulphonic acids of propylated or butylated aromatic hydrocarbons, particularly naphthalene derivatives, are added to dye liquors for the purpose of assisting wetting-out of the textile (wool) material and level dyeing. A. J. Hall.

Decorative printing of textile fabrics. Calico Printers' Assoc., Ltd., and A. Swallow (E.P. 261,448, 17.7.25).—Brocade effects are obtained by printing fabrics consisting of mixed fibres, *e.g.*, cotton and wool, or cotton and a suitable artificial silk, with a multi-coloured floral pattern in fit with a blotch pattern, the spaces between the two patterns being of sufficient width to prevent creeping of the printing pastes used. The blotch pattern is produced by printing the fabric with a caustic alkali capable of destroying one of the fibres (wool or artificial silk) forming the fabric. A. J. Hall.

Manufacture of ice colours. A. G. Bloxam. From Chem. Fabr. Griesheim-Elektron (E.P. 261,542) 19.11.25).—In dyeing yarns and fabrics by means of naphthol AS dyes (cf. E.P. 230,022; B., 1925, 878,

the formation of loose colour (not fast to rubbing) on the surface of the fibres is avoided by displacing the unabsorbed "prepare" solution by treating the fabric before development with a much weaker "prepare" solution containing sodium chloride. For example, yarn prepared with a solution containing 7 g. of naphthol AS is treated with a similar solution containing only 0·5 g. of naphthol AS, but also 30 g. of sodium chloride per litre. In many cases this method enables the usual formaldehyde in the original "prepare" solution to be omitted. A. J. HALL.

Obtaining lustre and matt effects on fabrics composed wholly or in part of viscose in the sulphur state. BURGESS, LEDWARD & Co., LTD., F. SCHOLEFIELD, and N. DENVER (E.P. 261,099, 14.8.25).—Lustre and matt effects are obtained by printing fabric containing non-desulphurised viscose silk with a paste containing sulphite of sodium, potassium, ammonium, or calcium, together with gum or glycerin or other suitable assistant, then steaming, rinsing, soaping, and drying; the printed parts are thereby desulphurised and rendered more lustrous. Sodium sulphide (cf. Scholefield, J. Soc. Dyers and Col., 1923, **39**, 342) is not suitable for use as a desulphurising agent because of its deleterious action on the usual copper printing rollers. Suitable direct, basic, and vat dyes may be added to the desulphurising paste. A satisfactory coloured printing paste contains 1·5 lb. of benzopurpurine, 10 lb. of glycerin, 1·5 lb. of sodium phosphate, and 10 lb. of sodium sulphite. A. J. HALL.

Apparatus for mercerising fabrics. L. MELLERSH-JACKSON. From MASCHINENFABR. BENNINGER A.-G. (E.P. 262,343, 24.6.26).

Improving cellulosic materials, in particular vegetable textile fabrics (E.P. 252,360).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Absorption of water vapour by sulphuric acid solutions. C. H. GREENEWALT (Ind. Eng. Chem., 1926, **18**, 1291—1295).—It is found that in the absorption of water vapour by sulphuric acid in a wet-walled tower the resistance of the stagnant film of liquid at the gas-liquid interface is negligible (cf. Lewis and Whitman, B., 1925, 55). By altering the degree of turbulence by using various shapes of entrance nozzles for the gas or introducing packing into the tower, and by varying the velocity of the carrier gas, it has been shown that for counter-current flow both these variables are proportional to the resistance to flow through the tower, in so far as they affect the overall gas-film diffusion coefficient. R. CUTHILL.

Mass law in the manufacture of nitric acid. C. W. CUNO (Ind. Eng. Chem., 1926, **18**, 1298—1299).—If the partial decomposition of nitric acid when prepared from sodium nitrate is due in part to the reaction $4HNO_3 = 2H_2O + O_2 + 2N_2O_4$ brought about by the dehydrating action of the sulphuric acid, introduction of an excess of oxygen or air into the retort might lessen the decomposition, and, in any case, the lower oxides of nitrogen would probably be in the most reactive state in the retort. Experiments show that the formation of brown fumes in the distillation can be very nearly prevented in this way, the amount of oxygen required increasing with the temperature of distillation. Cf. U.S.P. 1,552,117 (B., 1925, 847). R. CUTHILL.

Lime-kiln operation in the ammonia-soda process. J. KIRCHNER (Chem.-Ztg., 1926, **50**, 957—958).—In the ammonia-soda cycle the production of a satisfactory kiln-gas is more important than the quality of the lime. 10% above the theoretical quantity of carbon dioxide is needed owing to incomplete absorption. Loss also occurs in the recovery of carbon dioxide from the sodium bicarbonate, so that for the production of 100 kg. of soda ash 90 kg. of carbon dioxide must be supplied, 60% of this from the lime-kilns. From a calculation of gas volumes it is shown that the raising of the kiln-gas from 30% to 40% CO_2 saves 4 tons of coal per day, in a plant producing 100 tons of soda ash daily, in respect of economy in power in the gas compressors. A saving in ammonia also results. With a coke consumption of 9·5% in the kiln, a gas can in theory be produced containing 41·5% of CO_2. Increase in the coke ratio naturally produces a more dilute gas, but it is better to keep the gas concentrated, even if the limestone is incompletely burnt. The coke pieces should be of the size of the fist, and the limestone that of a child's head, as "smalls" produce irregularities in the gas flow, which should also be controlled by a fan. The kiln should be kept under pressure. The exit gases should not leave at a temperature above 150°, and the burnt lime should come out only hand-warm. Continuous charging is desirable. C. IRWIN.

Ammonia catalysts. A. T. LARSON and A. P. BROOKS (Ind. Eng. Chem., 1926, **18**, 1305—1307).—The catalytic activities of iron, tungsten, molybdenum, nickel, and cobalt in the synthesis of ammonia from its elements at 450°, 500°, and 550° and under a pressure of 30 atm. have been examined. Of these metals the last two are inactive, and iron is the most active, although its activity falls off very rapidly, especially at the higher temperatures. Mixtures of molybdenum with iron, cobalt, and nickel are slightly less effective. The addition of silica, thoria, alumina, zirconia, or ceria increases the activity of iron. Mixtures of oxides are most effective in this way when the constituent oxides are not of the same type; thus an iron catalyst containing 1% of potassium oxide and 3% of alumina is specially recommended. R. CUTHILL.

Pure iron and promoted iron catalysts for ammonia synthesis. J. A. ALMQUIST and E. D. CRITTENDEN (Ind. Eng. Chem., 1926, **18**, 1307—1309).—Iron is most active in catalysing the synthesis of ammonia if it has been prepared by reduction of ferrosoferric oxide. If the original oxide contained an excess of ferrous oxide, there is a continuous decrease in activity with increase in the amount of this component. Potassium oxide appears to depress the activity of iron, unless some other refractory oxide such as silica is also present. Further evidence of the marked "promoting" effect of mixtures of potassium oxide with difficultly reducible amphoteric oxides such as silica and alumina has been obtained (cf. Larson and Brooks, preceding abstract; also B., 1922, 325 A). R. CUTHILL.

Simple apparatus for the removal of ammonium salts by volatilisation. K. Fischer (Z. angew. Chem., 1926, 39, 1574—1576).—An apparatus for volatilising ammonium salts from residues, such as are obtained in the analysis of the alkali metals, consists of a metal funnel from which the stem has been removed and which is lined inside with a clay tube in the shape of a truncated cone. The inside of the clay tube is covered with iron wire gauze, which also closes the hole at the lower end, at which point it is covered with a perforated metal plate. The basin containing the salts is placed inside the top of the gauze funnel, and is heated by means of a Bunsen burner, the flame of which is at least 12 cm. long when the gas is full on. The top of the burner is 1 cm. below the perforated plate, so that by the use of a small flame the solution may be evaporated to dryness without spirting, and subsequently the ammonium salts are volatilised by the use of the full flame of the burner. A. R. Powell.

Distillation of calcium acetate. E. G. R. Ardagh, A. D. Barbour, G. E. McClellan, and E. W. McBride (Fac. Appl. Sci., Toronto, 1926, Bull. 6, 85—104).—Calcium acetate, made by adding a slight deficiency of calcium carbonate to acetic acid and evaporating to dryness, does not become completely anhydrous even after prolonged heating at temperatures below 160°. After 12 hrs. at 160° the salt still contains 2% of free acetic acid; at higher temperatures decomposition takes place and traces of acetone begin to be evolved, but a temperature above 400° is necessary for the rapid production of acetone. In order to obtain a high yield of this compound the vapours in the retort must be removed as rapidly as they are formed, preferably by means of a current of nitrogen or carbon dioxide; air should not be used as it reduces the yield considerably. A nearly theoretical yield is obtained at 450—490° in a good current of gas, and the presence of iron or copper has no deleterious effect; at 430° the maximum yield is 93%, and at 420° only 85%. A. R. Powell.

Behaviour of difficultly soluble metal oxides in solutions of their salts. Magnesium oxide. I. W. Feitknecht (Helv. Chim. Acta, 1926, 9, 1018—1049).—The behaviour of magnesium oxide in solutions of magnesium chloride, bromide, sulphate, and nitrate has been studied in detail, with special reference to the effects of concentration, temperature, method of preparation of the oxide, and other influences on the velocity of the formation of the basic salt, and the composition and crystalline form of the product, the examination being carried out by viscosimetric and volume-change measurements, and microscopical examination of the precipitates formed. Four types of magnesium oxide were employed, these being prepared, respectively, from magnesium carbonate at 600° (*a*) and 1100° (*b*), and from magnesium oxalate at the same temperatures (*c* and *d*). The main results obtained are as follows: the solubility of magnesium oxide in magnesium chloride solutions increases with rise of temperature and with increase in the concentration of the salt solution, and in supersaturated solutions the velocity of formation of the basic chloride is more rapid the higher the temperature and the greater the degree of supersaturation, and for each temperature there exists a definite concentration below which, instead of the basic chloride, magnesium hydroxide separates, and this concentration increases with rise in temperature. In solutions of magnesium chloride containing an excess of magnesium oxide in suspension the behaviour of the mixture depends very largely upon the concentration of the chloride solution and the sample of oxide used. In general, in solutions of sufficient concentrations for any change to occur, the basic salt separates first in an amorphous form, which then slowly changes into a crystalline form, the velocity and duration of the reaction depending on the factors mentioned. In dilute solutions, *i.e.*, in concentrations below which any change in the form of the basic salt occurs, the reaction starts sooner, and there is originally a large swelling, accompanied by an increase in the viscosity of the medium to a maximum value, which then disappears, the viscosity falling again. The amount of this swelling is greater with sample (*a*), which is very disperse, than with the more compact sample (*c*). Heating the oxide to 1100° causes a large diminution in the surface area, and the reaction velocity is thereby decreased, the adsorption complex being slowly formed and going slowly into solution, so that the basic salt separates slowly in large crystalline form. In more concentrated solutions the viscosity increases more slowly to a greater maximum value, and in higher concentrations (7—8N) of the chloride the whole reaction mixture becomes stiff. The value of the concentration below which no change in form occurs is usually considerably higher than the concentration at which the basic chloride separates from supersaturated solutions, the two being more nearly identical with samples (*b*) and (*d*), and it increases rapidly with rising temperature. The ratio of oxide to chloride directly affects mainly the rate at which the reaction sets in. Magnesium hydroxide reacts much more slowly than the oxide, the velocity still being immeasurably small in relatively concentrated solutions. The basic salt formed was separated and analysed in each case; its crystalline form and chemical composition were found to depend upon the sample of oxide used, the concentration of the solution, and the temperature. The proportion of magnesium oxide was found, in general, to be greater the more dilute the solution, the value of the ratio $MgCl_2/MgO$ in the product varying approximately from 1/2·5 to 1/5. The water content of the product, dried over phosphorus pentoxide, corresponds to 10 mols. for each mol. of magnesium chloride. The product obtained from 3·5N- and 4N-solutions agreed with the composition $MgCl_2,5MgO,10H_2O$; that from 3N-solutions with $MgCl_2,4MgO,10H_2O$; that from 5N-solutions, and when magnesium hydroxide was used in place of the oxide, with $MgCl_2,3MgO,10H_2O$; and that from 6N- and 8N-solutions with $2MgCl_2,5MgO,16H_2O$, but long contact with the mother-liquors alters both the crystalline form and the composition of the product. Similar results were obtained in the other magnesium salt solutions. J. W. Baker.

Fixation of nitrogen as aluminium nitride. H. J. Krase, J. G. Thompson, and J. Y. Yee (Ind. Eng. Chem., 1926, 18, 1287—1290).—Preliminary experiments in which mixtures of bauxite, coke, and magnetite

were heated in an electric furnace showed that, although reduction was incomplete, alloys containing 40% or more of aluminium were formed. If a ferroaluminium containing 50% Al is heated at 1400° in nitrogen under atmospheric pressure, about 60% of the aluminium is converted into nitride. The presence of small amounts of magnesium promotes nitrification, whilst calcium, silicon, and titanium have the opposite effect. If small quantities of cryolite, or of the chlorides or fluorides of aluminium, magnesium, sodium, or calcium are present, nitrification may be rapid and practically complete even at 1200°. With ferrotitanium or aluminium carbide at 1400° the absorption of nitrogen is only about 20% of the theoretical, whilst for ferrosilicon the figure is only 2%. R. CUTHILL.

Variations in density of the atmosphere. A STOCK and G. RITTER (Z. angew. Chem., 1926, **39**, 1463—1464).—This preliminary communication shows that the common belief in the constancy of the density and composition of air free from moisture and carbon ioxide is in error. With the aid of a special air-density balance (A., 1926, 669) it is demonstrated that the variations in density may be as much as 0·13% even in rural surroundings. This corresponds to a variation of 0·75% of oxygen, a matter which it is proposed to examine further. E. HOLMES.

PATENTS.

Manufacture of [concentrated] oleum. J. H. SHAPLEIGH, Assr. to HERCULES POWDER Co. (U.S.P. 1,605,004, 2.11.26. Appl., 3.2.25).—Gases from a converter containing about 7% SO_3 are cooled, and passed into 20% oleum, to which 98% sulphuric acid is added to balance the sulphur trioxide absorbed. The gases are then led into a second absorber, where they are treated with water to remove the residual sulphur trioxide with formation of 98% sulphuric acid, which is employed in the first absorber. The heat evolved in the formation of the 98% acid is used to distil pure sulphur trioxide from a portion of the 20% oleum, which is then returned to the first absorber, and the evolved gas is passed into a further quantity of 20% oleum to form concentrated oleum of any desired strength. T. S. WHEELER.

Manufacture of sulphuric acid. E. R. WOLCOTT, Assr. to THE TEXAS Co. (U.S.P. 1,605,088. 2.11.26. Appl., 4.12.24).—Sulphur dioxide and oxygen are passed over heated diaspore to produce sulphur trioxide. H. ROYAL-DAWSON.

Absorbing sulphur trioxide from gases containing same. J. H. SHAPLEIGH, Assr. to HERCULES POWDER Co. (U.S.P. 1,608,006, 23.11.26. Appl., 22.4.25).—Sulphuric acid is sprayed on the contact gases, countercurrent to the gas flow and at a number of points along the path of the gases passing through a horizontal cylinder. By this means a progressive reduction in the sulphur trioxide vapour pressure is produced at certain points of absorption, and acid of progressively decreasing strength is continuously produced. The separate products are collected within the cylinder. H. ROYAL-DAWSON.

Manufacture of silicic acid gel. W. CARPMAEL. From I.-G. FARBENIND. A.-G. (E.P. 262,306, 19.2.26).—Silicic acid jelly is washed, or dehydrated by pressure, prior to treatment with sodium carbonate solution, or with ammonia, water glass, and the like, the product being subsequently dried. H. ROYAL-DAWSON.

Production of moulded silicic acid gel. M. PRÄTORIUS and K. WOLF (E.P. 249,555, 19.3.26. Conv., 21.3.25).—A silicic acid jelly containing 75—95% of water is mixed with 1—2% of a colloidal silicic acid solution of 1—10% strength, the homogeneous mass being then moulded and dried at 110—180°. A modification of the process is obtained by using the silicic acid jelly itself and gradually heating it under pressure at 100—300°, the mass being then moulded and dried as before. H. ROYAL-DAWSON.

Manufacture of hydrocyanic acid. J. Y. JOHNSON. From I.-G. FARBENIND. A.-G. (E.P. 261,559, 14.12.25).—Formamide, alone or mixed with ammonium formate, is rapidly heated between 400° and 900° in a quartz tube in the absence of other gases. The reaction may be carried out in the presence of various dehydrating catalysts, *e.g.*, alumina, bauxite, titanium oxide, silica gel, etc., supported with pumice, asbestos, or active charcoal, the resulting hydrocyanic acid being rapidly cooled. H. ROYAL-DAWSON.

Production of hydrocyanic acid. B. S. LACY, Assr. to THE ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,608,700, 30.11.26. Appl., 20.2.26).—In the thermal decomposition of formamide in the presence of a catalyst capable of causing the formation of hydrocyanic acid and water, the formamide vapour is mixed with a large volume of an inert heating gas at above 500° before it comes in contact with the catalyst. H. ROYAL-DAWSON.

Process for oxidising a ferrous salt. R. F. GRANT and E. WETHERBEE, Assrs. to H. M. HANNA (U.S.P. 1,606,470, 9.11.26. Appl., 9.3.25).—A subdivided catalyst (manganese dioxide) is introduced into a ferrous sulphate solution, which is circulated in contact with it in the presence of air. H. ROYAL-DAWSON.

Producing pure aluminium hydroxide, particularly from aluminium-containing raw materials rich in silica. E. L. RINMAN (E.P. 243,356, 18.11.25; Conv., 22.11.24).—The sulphate solution, after first treating the raw material with sulphuric acid, is mixed with a hot solution of sodium hydrosulphide containing sodium sulphide to precipitate the aluminium hydroxide and liberate hydrogen sulphide. The aluminium hydroxide obtained is dissolved in sodium sulphide solution, precipitated with hydrogen sulphide, filtered, and washed. H. ROYAL-DAWSON.

Method of producing pure aluminium hydroxide. G. H. HULTMAN (U.S.P. 1,607,279, 16.11.26. Appl., 8.5.25. Conv., 25.1.24).—Aluminium mineral is heated with sulphuric acid producing a solution of the sulphate in which the iron present is reduced to the ferrous state; on addition of calcium carbonate to the solution, the ferrous salt, undissolved aluminium hydroxide, and calcium sulphate are separated and washed with water to dissolve out the calcium sulphate. H. ROYAL-DAWSON.

Manufacture of chromates. W. CARPMAEL. From I.-G. FARBENIND. A.-G. (E.P. 261,647, 25.6.26).—A mixture containing ground chrome iron ore, sodium car-

bonate, ferric oxide, and a small quantity of magnesia, is introduced in the wet or dry state into a mechanical furnace and heated above 1000°, after which the melt is quenched in hot water to dissolve out the aluminium salts. The residue is dried, ground, and again brought to the desired composition with ferric oxide or magnesia and re-used with fresh material. The alumina is precipitated from the chromate liquor by waste gases or acids. H. ROYAL-DAWSON.

Separating aluminium nitrate from mixed solutions of potassium, sodium, iron, calcium, and magnesium nitrates. S.I.P. Soc. ITALIANA POTASSA (E.P. 246,827, 18.1.26. Conv., 28.1.25).—Nitric acid is added to the unevaporated mixed solution of these salts in such a manner as to produce the crystallisation of aluminium nitrate, which is complete according to the degree of acidity, while the remaining nitrates remain in solution. H. ROYAL-DAWSON.

Process for producing phosphorus pentoxide. C. E. PARSONS and S. PEACOCK, Assrs. to METAL RESEARCH CORP. (U.S.P. 1,606,319, 9.11.26. Appl., 29.3.24).—The blast furnace is charged with material containing calcium phosphate, silica, coke, and an alkali chloride. H. ROYAL-DAWSON.

Process and apparatus for making sodium chloride. H. T. REID (U.S.P. 1,605,375, 2.11.26. Appl., 19.2.24).—The top surface of the salt solution is heated uniformly with hot air without causing ebullition or disturbance of the solution, which is maintained at a fixed level. H. ROYAL-DAWSON.

Production of potassium salts [from sea water]. E. NICCOLI (E.P. 261,991, 13.5.26; cf. F.P. 594,904 and E.P. 247,405; B., 1926, 237, 360).—The process described in F.P. 594,904 is modified so that instead of dissolving the mixture of sodium, potassium, and magnesium salts in water to form a saturated solution, the salts are treated with a quantity of water insufficient to give complete solution, whereby the sodium salts and chlorides are dissolved, leaving the double salt of potassium and magnesium sulphate as a residue. W. G. CAREY.

Obtaining sulphur from alkaline-earth sulphates. SALZWERK HEILBRONN A.-G., T. LICHTENBERGER, and K. FLOR (E.P. 251,942, 29.1.26).—Carbon in the form of coke is added to alkaline-earth sulphate dissolved in molten sodium chloride, and, after reduction, the alkali chloride is separated from the alkaline-earth sulphide by sedimentation, the latter being converted, by treatment with vapour or acid, into the oxide or salt and hydrogen sulphide, from which sulphur is obtained in the usual manner. H. ROYAL-DAWSON.

Purifying and concentrating sulphur dioxide. G. C. HOWARD (U.S.P. 1,606,299, 9.11.26. Appl., 3.4.23).—In the absence of water and phenols, a liquid cyclic hydrocarbon forms compounds with sulphur dioxide, which can thus be removed from a dry gas containing it. The hydrocarbon and the sulphur dioxide are separated and recovered by heating. R. A. A. TAYLOR.

Manufacture of carbon disulphide. A. J. STEPHENS. From ZAHN & Co. BAU CHEM. FABR. G.M.B.H. (E.P. 261,990, 12.5.26).—Raw sulphur is freed from impurities in a melting apparatus comprising two parts, so that impurities can be removed by a siphon device at one side, while the other part is in use. The fluid sulphur is led to a retort constructed so that the lower part is broadened and ribbed to increase the heating surface; the retort contains wood charcoal, and the mixture is heated from the outside. The crude carbon disulphide is distilled, and on cooling much of the hydrogen sulphide present as an impurity escapes and is led to a specially constructed Claus furnace for conversion into sulphur for use again in the process. The last traces of hydrogen sulphide are removed by spraying the carbon disulphide into a separator containing pure, nearly boiling carbon disulphide, the escaping hydrogen sulphide being freed from carbon disulphide vapour in the cooler and conveyed to the Claus furnace, while the carbon disulphide is drawn off from the separator by a siphon, and is cooled and stored. The residue in the distilling vessel, consisting of carbon disulphide rich in dissolved sulphur, is drawn off periodically into a funnel-shaped container with ribbed walls, strongly heated on all sides, in which the carbon disulphide evaporates and is sent to the cooling system, after which the sulphur melts and is run back through a heated siphon to the retort. W. G. CAREY.

Separation of hydrocyanic acid from gaseous mixtures containing it. O. LIEBKNECHT, Assr. to DEUTSCHE GOLD- & SILBER-SCHEIDEANSTALT VORM. ROESSLER (U.S.P. 1,605,897, 2.11.26. Appl., 9.8.24. Conv., 1.8.23).—See G.P. 403,378; B., 1925, 205.

Electrolyser for the production of hydrogen and oxygen (E.P. 242,635).—See XI.

Preparation of titanium compounds [for pigments] (E.P. 261,051).—See XIII.

Producing a titanium pigment [titanium oxide] (U.S.P. 1,605,851).—See XIII.

VIII.—GLASS; CERAMICS.

Lubricants for ground-glass joints. M. J. BRADLEY and H. E. WILSON (Ind. Eng. Chem., 1926, **18**, 1279—1280).—A number of lubricating mixtures are described, including one which is very resistant to the action of strong reagents. Heavy liquid paraffin oil is superior to vaseline as a base. R. CUTHILL.

Effect of porosity upon thermal conductivity, diffusibility, and heat capacity at high temperatures. Y. TADOKORO (Sci. Rep. Tôhoku Imp. Univ., 1926, **15**, 567—596).—Refractory materials were prepared from a mixture of 2 pts. (by vol.) of alundum powder (80 mesh/in.) and 1 pt. of washed "Gairome" fireclay, and the porosity was varied by adding increasing amounts of sawdust. The properties of these materials were then examined, and also of specimens made from diatomaceous earth. The porosity and gas permeability vary similarly. The permanent changes in weight and length caused by heating at temperatures up to 1300° were measured; if diatomaceous earth is heated to 1100° before use, it can subsequently be heated at high temperatures without fear of cracking from volume change. Both thermal expansion and crushing strength decrease with increasing porosity, the curve connecting these constants resembling a rectangular hyperbola.

If K is the thermal conductivity, ρ the sp. gr., and C the sp. heat, the diffusibility h^2 is given by $h^2 = K/\rho c$. The effects of porosity on diffusibility, thermal conductivity, and heat capacity have been determined; in all cases a minimum value is given by a porosity of 37—38%. The spalling tendency is expressed by the relation ($\alpha \times$ crushing strength)/h, where α is the coefficient of expansion; here, again, a porosity of 37—38% is a critical point which is of importance in the manufacture of fire-bricks. Diatomaceous earth is a very good insulating material, but its crushing strength is low.

W. HUME-ROTHERY.

Modulus of rupture of dried clays as a measure of plasticity. H. KOHL (Ber. Deuts. Keram. Ges., 1926, 7, 19—31).—Previous investigations have indicated a certain relation between the mechanical strength and the bonding strength of clays. The modulus of rupture test, in preference to other mechanical tests, was chosen to give an indication of plasticity because of its simplicity in operation, and because the results give a more uniform and reliable indication of the chief factor in plasticity, viz., the strength of the bond in clays. The tests were carried out on air-dried specimens of clays and body mixtures. Blue clays gave the highest results—47 kg./sq. cm.—and fine kaolins the lowest—2·8 kg./sq. cm. For practical purposes, the minimum strengths, in kg./sq. cm., are: for earthenware, 7—8; for porcelain, 4—5; and for saggars, 5—7. The addition of alkali hydroxides to casting slips almost doubled the dry strength as compared with specimens moulded from the same body. Cast samples were also more dense than moulded. The results of the modulus of rupture tests were characteristic of the different mixtures tested, and agreed reasonably well with the working properties as observed in actual practice.

F. SALT.

Effect of industrial usage on thermal conductivity of semi-silica material used in a coke-oven wall. A. T. GREEN (Trans. Ceram. Soc., 1925—6, **25**, 258—261).—See B., 1926, 947.

PATENTS.

Manufacture of glass and articles therefrom. E. A. HAILWOOD (E.P. 261,491, 1.9.25).—Molten glass of high fluidity, *e.g.*, a glass prepared from 150 pts. of sand, 84 pts. of sodium carbonate, 42 pts. of borax, 6 pts. of magnesia, 6 pts. of zinc oxide, and 12 pts. of calcium carbonate spar, is cast in sand, plaster, or metal moulds, and annealed either in the moulds or after removal therefrom.

B. W. CLARKE.

Method and apparatus for purifying quartz. BRITISH THOMSON-HOUSTON CO., LTD., Assees. of E. THOMSON (E.P. 252,747, 1.6.26. Conv., 1.6.25).—Silica is fused at 1650—1700° in an electrically-heated crucible rotating at 1500 r.p.m., so that the lighter portion of the melt containing entrapped gases is segregated from the remainder, which upon solidifying forms a homogeneous mass of clear quartz.

B. W. CLARKE.

Pottery and other kilns. MORGAN CRUCIBLE CO., and C. W. SPEIRS (E.P. 261,866, 9.9.25 and 18.12.25).—In a tunnel kiln the firing zone is heated by one or more electrical resisters (E.P. 225,608), forming either the roof alone, the roof and sides, or placed centrally in the tunnel. Flues may be provided for burning oil, gas, or waste gases, so as to minimise radiation of electrically-developed heat. Other modifications are (1) an endless circular tunnel, (2) a sloping tunnel with a raised firing zone, (3) suitable heating of other zones of the kiln by resisters, (4) regulation to give an oxidising or reducing atmosphere as required.

A. COUSEN.

Manufacture of grinding or abrading stones. E. MENZIONE and L. MAYER (E.P. 261,942, 19.1.26).—The stones are made from a paste consisting of burnt magnesite, graphite, minium, steel filings, and corundum or carborundum, together with hydrochloric acid. The paste, reinforced with wire netting, is moulded and dried by pressure. It is finally treated with a solution of magnesium fluosilicate at about 80° and then dried.

A. COUSEN.

Neutral refractory cement. W. F. ROCHOW, Assr. to HARBISON-WALKER REFRACTORIES CO. (U.S.P. 1,606,481, 9.11.26. Appl., 20.1.23).—A refractory composition consists of an intimate mixture of powdered aluminium oxide and powdered sodium silicate.

B. W. CLARKE.

Dental cements. S. S. WHITE DENTAL MANUFACTURING CO., Assees. of N. E. EBERLY (E.P. 248,698, 11.8.25. Conv., 3.3.25).—A dental cement consists of from 70 to 98% of a basic glass, prepared by fusing together silica and alumina in the presence of a suitable flux, *e.g.*, calcium fluoride, which is powdered and mixed with a powder containing basic oxides, *e.g.*, zinc oxide and silica, bismuth oxide, etc. Aqueous phosphoric acid containing phosphates of zinc, aluminium, etc. in solution is added to give the required consistency.

B. W. CLARKE.

Manufacture of sheet glass. E. C. R. MARKS. From ERIE GLASS CO. (E.P. 261,150, 28.9.25).

Apparatus for forming sheet glass. L. MELLERSH-JACKSON. From HARTFORD-EMPIRE CO. (E.P. 261,627, 31.5.26).

Means for making gas-tight joints between metal and glass or quartz (E.P. 243,000).—See X.

Sealing metal to glass (E.P. 251,951).—See X.

Sealing molybdenum or similar refractory metals and fused quartz (U.S.P. 1,608,612).—See X.

IX.—BUILDING MATERIALS.

Lime-kiln operation in the ammonia-soda process. KIRCHNER.—See VII.

PATENTS.

Manufacture of cement. T. RIGBY (E.P. 261,814, 24 and 25.5.26, and 12.8.25).—In the manufacture of cement by the wet method in a rotary kiln, a higher kiln output is attained by spraying the slurry into the kiln, thus reducing its water content to about 10% before it is deposited on the wall, and preventing the formation of "slurry rings" or large masses. One or more sets of long sprays are employed, originating outside the kiln, and disposed at such an angle to the axis that even if projected several feet into the kiln the spread is not greater than the internal diameter of the kiln, so that the still moist slurry is gently deposited upon the wall. Slurry lifting or agitating gear is provided

internally only beyond the region where the atomised material is mainly deposited, and a scraping device is added which, during rotation, frees the kiln mouth from deposit. W. G. Carey.

Calcining and clinkering cement-forming materials. R. D. Pike (U.S.P. 1,605,279, 2.11.26. Appl., 13.6.23. Cf. U.S.P. 1,468,168; B., 1923, 1073).—The material to be treated is passed through a calcining chamber and a clinkering chamber, and the hot material is then agitated in a cooling chamber, through which is passed substantially the whole of the air needed to support combustion in the other two chambers, in order to absorb the sensible heat from the hot clinker. B. W. Clarke.

Treating cement mix and other materials. J. W. Hornsey, Assr. to Granular Iron Co. (U.S.P. 1,606,125, 9.11.26. Appl., 9.7.19. Renewed 26.1.25).—Cement mix is calcined in a rotary kiln supplied with fuel in fixed proportions, and arranged so that the egress of gases from the furnace end is prevented, and the total air supply at the furnace end relative to the draught outlets at the stack end is restricted until the oxygen supply is practically limited to the theoretical requirement of the fuel used. B. W. Clarke.

Utilising heat from cement clinker. W. S. Speed, Assr. to Louisville Cement Co. (U.S.P. 1,606,850, 16.11.26. Appl., 23.3.20).—Clinker from rotary or other types of kilns is fed into a closed chamber, and air is passed backwards and forwards through the clinker the air then being used as a heating medium. W. G. Carey.

Ageing or seasoning cement. J. W. Fuller (E.P. 250,599, 8.4.26. Conv., 9.4.25).—Freshly-ground cement is discharged into a cement pump consisting of a cylindrical barrel in which a differential screw rotates at high speed, and moist gases, having a high carbon dioxide content from the products of combustion in the cement kiln, are injected through orifices at high pressure into the cement. The moist gas, in intimate mixture with the cement, converts any free lime into carbonate and hydroxide, and also causes the cement to flow more easily. Cement so treated can be used almost immediately after grinding. W. G. Carey.

Process for ageing or seasoning cement. J. W. Fuller (U.S.P. 1,608,499, 30.11.26. Appl., 9.4.25).—See E.P. 250,599; preceding.

Preservation of stone. A. P. Laurie (U.S.P. 1,607,762, 23.11.26. Appl., 31.10.25. Conv., 29.8.23). —See E.P. 221,342; B., 1924, 911.

Concrete mixing apparatus. Ransomes & Rapier, Ltd., and P. D. Ionides (E.P. 261,910, 23.11.25).

Grinding members for ball, drum, and tubular mills [for Portland cement] (E.P. 261,664).—See I.

Process and apparatus for electrodeposition [for "copperclad shingles"] (E.P. 238,230).—See XI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Dendritic crystallisation and grain formation in steels. V. N. Krivobok (Trans. Amer. Soc. Steel Treat., 1926, **10**, 758—781).—The characteristics of dendrites are considered. Photomicrographs are given of the structure of dendritic skeletons from low- and high-carbon steels which show that on cooling a single dendrite is changed into a number of grains. The causes of granulation may include changes in structure accompanying change in atomic arrangement at the A3 point, conditions of strain existing in the ingot after solidification, and the presence of mechanically-held inclusions. The grain size in castings does not depend on the size of the original dendrites. Large dendrites accompany slow cooling, which favours the formation of inclusions in patterns and pronounced segregation. The formation of banded structure in hot-rolled blooms is due to the same cause. T. H. Burnham.

Chromium–cobalt steels. F. M. Ostraga (Compt. rend., 1926, **183**, 882—885).—The effect of a second heating, after a primary heating to temperatures between 900° and 1100°, has been studied for two chromium–cobalt steels (composition given), both dilatometrically and micrographically. It is concluded that they are at least as sensitive as, if not more so than, high-speed chromium–nickel steels to the temperature of heating and the rate of cooling. Indications are obtained that an austenite–troostite transformation takes place in the steels after heating them to a point just above their transition temperature, and slowly cooling. S. J. Gregg.

Forging by the upset process. J. C. Kielman (Trans. Amer. Soc. Steel Treat., 1926, **10**, 599—614).—Ball-bearing rings are forged by upsetting bars of steel containing 0·95—1·10% C and 1·20—1·50% Cr, using tungsten steel dies. The pierced-out centre is left on the end of the bar and used in the next forging. The forging temperature is approximately 1100°. The flow of metal in the forgings is shown by an etching process. The wear of the dies does not seem to depend on their ball hardness, and the temperature had a greater effect than the pressure. The oil-fired furnaces for heating the bars are described. T. H. Burnham.

Behaviour of industrial copper during cold working. E. Seidl and E. Schiebold (Z. Metallk., 1926, **18**, 241—246, 315—321, 343—346).—When ordinary commercial copper is cast into ingots in open moulds crystallisation takes place in such a way that the cross-section of the ingot may be divided into several well-marked zones according to the crystal structure; the outer surface layer immediately against the walls of the mould consists of a heterogeneous mixture of very small crystals with a large proportion of the impurities, especially cuprous oxide, next there is a broad layer of large fibrous crystals arranged perpendicular to the walls of the mould, and, finally, the interior is built up of three distinct zones, the lower consisting of small "seed" crystals, the middle of larger, more regular crystals, and the upper of somewhat smaller, unevenly oriented crystals forming generally a somewhat porous mass owing to inclusions of gas. Pores may also be discerned along the boundaries of the fibrous crystals, between the fibrous layer and the central zones, and, to a much smaller extent, in the mass of large crystals in the centre of the ingot. Besides exhibiting differences of macro-

structure the zones also show characteristic X-ray diffraction patterns, and the hardness and mechanical properties vary considerably, the fibrous zones being generally the toughest and the surface skin the hardest. During cold rolling the surface layers usually develop fissures which on severe rolling tend to penetrate the middle layers; after removal of the surface skin, however, the remainder of the ingot may be rolled without danger of fracture, although sometimes the central zone will develop cracks after a 60% reduction in thickness. The presence of cuprous oxide or gas inclusions between the fibrous crystals will also sometimes cause splitting when they are rolled along the longest diagonal. Prolonged annealing of the cast bars at 750° results in a more homogeneous structure, but causes the separation of relatively coarse particles of cuprous oxide. The only satisfactory procedure for eliminating troubles in rolling consists of ensuring more homogeneous castings by avoiding oxidation of the metal as much as possible during casting and by pouring quickly in a steady stream at the correct casting temperature into thick-walled moulds. A uniformly fine structure may then be obtained by forging the ingot to reduce its cross-section by 5—10% and subsequently annealing at 750° for 1 hr. previous to rolling. A. R. POWELL.

Equilibrium between zinc vapour, carbon monoxide, and carbon dioxide. E. THEIS (Z. angew. Chem., 1926, **39**, 1568—1574).—The equilibrium of the reaction $ZnO + CO = Zn + CO_2$ is given by the equation $\log(p_{Zn}\cdot p_{CO_2}/p_{CO}) = -10{,}100/T + 9\cdot62$ where p_{Zn}, p_{CO}, and p_{CO_2} are the partial pressures of zinc vapour, carbon monoxide, and carbon dioxide, respectively, at a temperature of $T°$ Abs. The total vapour pressure and the partial pressures of zinc vapour, carbon monoxide, and carbon dioxide over a heated mixture of graphite and zinc oxide at 660—880° have been calculated from this equation; the results agree fairly well with those of Maier and Ralston (A., 1926, 358) and indicate that in the presence of liquid zinc the total vapour pressure reaches 1 atm. at above 870°. A. R. POWELL.

Structure and properties of red brass. R. KÜHNEL (Z. Metallk., 1926, **18**, 273—278, 306—311).—Red brass alloys containing 80—85% Cu, 4—9% Sn, and 9—4% Zn, with or without the addition of 2—3% Pb, are used as bearing metals on the German State Railways, the usual alloy containing 85% Cu, 9% Sn, and 6% Zn. Occasionally brittleness has developed, and this is shown to be due either to the presence of sulphur, as little as 0·02% of which causes the segregation of a sulphide constituent in the grain boundaries, or to inverse segregation. The latter is especially marked when about 2·5% Pb is added to the alloy and when dry sand moulds are used; in these cases the tin and lead contents of the outer shell of the casting may each be up to 0·5% above that of the core. The phenomena causing inverse segregation are discussed with reference to the work of other investigators, and experiments are described in which the physical and mechanical properties of many other alloys of the same type have been determined in an endeavour to find a cheaper alloy having similar properties to the above-mentioned alloy, but which has not such a tendency to inverse segregation. The most satisfactory alloy contained 82·8% Cu, 5·7% Sn, 9% Zn, and 2·3% Pb; it showed less wear with light loads than the 9% Sn alloy, but slightly more wear with heavy loads, otherwise its mechanical properties closely resembled those of the 9% Sn alloy. A. R. POWELL.

Structure of red brass [copper-tin-zinc alloys]. M. HANSEN (Z. Metallk., 1926, **18**, 347—349).—From the results obtained by the author in earlier work, and from those obtained by numerous other investigators, the ternary diagram of the system copper-tin-zinc has been constructed for alloys containing more than 50% Cu, and, on this as a base with vertical temperature ordinates, a space model has been constructed to show the equilibrium diagram of these alloys for temperatures up to 1100°. With reference to this model the behaviour of certain alloys of the system during cooling is discussed. A. R. POWELL.

Sand-cast aluminium-magnesium silicide alloys. S. DANIELS (Ind. Eng. Chem., 1926, **18**, 1280—1285; cf. B., 1926, 494).—The properties of alloys of aluminium with up to 13·5% of magnesium silicide have been examined. Alloys containing 1·25—1·75% of the silicide manifest, when quenched and artificially aged, both strength and ductility in a high degree. Annealing causes reduction in both strength and hardness compared with the sand-cast material, but increase in ductility. R. CUTHILL.

Determination of metallic lead in metallurgical products and pigments. D. H. MCINTOSH (Ind. Eng. Chem., 1926, **18**, 1320—1321).—The material to be analysed is boiled with a 15% solution of sodium hydroxide, which dissolves most of the lead salts, and the solution is then filtered. All the metallic lead remains in the residue, and is dissolved out with a 10% solution of silver nitrate, from which it is reprecipitated with sulphuric acid. Finally the lead sulphate is dissolved in a hot saturated solution of ammonium acetate, and titrated with standard ammonium molybdate solution, using tannic acid as indicator. R. CUTHILL.

Structure of electro-deposited metals. F. FOERSTER and J. FISCHER (Z. Elektrochem., 1926, 32, 525—534).—The dependence of the structure of electro-deposited tin on various factors, particularly on the nature and structure of the cathode material, has been examined. An electrolyte containing 0·25 mol. of stannous sulphate, 0·15 mol. of sulphuric acid, and 0·16 mol. of cresolsulphonic acid per litre (cf. Stack, B., 1924, 521), yields smooth, regular, thick deposits of tin at 0·25—0·5 amp./dm.^{2} Considerably higher concentrations of cresolsulphonic acid cause very soft, dull grey deposits unless the sulphuric acid concentration is correspondingly raised. Cresolsulphonic acid prepared from pure *m*-cresol acts much better than that from commercial cresol. Sulphonic acids derived from phenol, β-naphthol, naphthalene, or α-naphthylamine may also be used. Using a cathode of bright tin-plate in the electrolyte specified, the deposit has an appearance like that of tin-plate which has been etched with acid ("frost-flower" structure), the deposited tin atoms having arranged themselves so as to continue the lattices of the crystals forming the cathode surface (cf. Blum and Rawdon, B., 1923, 405; Graham, *ibid.*, 984). But at a cathode

of tin-plate roughened with emery or of tin foil, or at cathodes of other metals, tin is deposited under similar conditions in a fine-grained form, small crystals developing from many new nuclei. Whereas with deposits of the former kind, variations of current density, temperature, or electrolyte composition within wide limits produce little change in structure, with those of the latter type notable modifications may be produced similar to those observed in the deposition of copper or iron. On continued electrolysis, however, deposits of the "frost-flower" type eventually become overgrown with a fine-grained deposit; the higher the current density and the lower the tin content of the solution, the sooner this change sets in. The "frost-flower" structure of deposits on tin-plate can also be obtained from other tin baths capable of yielding smooth deposits, *e.g.*, from sulphostannate solutions or from a strongly acidified stannous sulphate solution containing 1% of gelatin. Addition of *m*-cresol itself to a strongly-acidified stannous sulphate bath restrains the formation of outgrowths, whilst the addition of 5—10 c.c. of *m*-cresol and 0·5 g. of gelatin to a litre of such a bath gives at 11° and 0·5 amp./dm.^{2} an extremely fine-grained, silvery deposit suitable for plating purposes. Deposition of cadmium from faintly acid cadmium sulphate solutions on coarsely crystalline cadmium surfaces also results in the continued growth of the crystals forming the cathode surface. Cresolsulphonic acid has no effect in this case, but it suppresses the formation of outgrowths in the deposition of cadmium from ammoniacal cadmium solutions. The addition of cresolsulphonic acid to acid stannous sulphate solutions does not notably change the stannous-ion concentration, nor does it appear to alter the nature of the cathodic deposition process, for neither the equilibrium potential of tin in the solution nor the cathodic polarisation is much changed. Its possible behaviour as a colloid might account for the suppression of outgrowths, but not for its favourable influence on the formation of "frost-flower" structure, which only forms when colloids are reduced to a minimum. The anion of cresolsulphonic acid probably acts in a similar manner to that of the anions ClO_4', BF_4', and SiF_6'' in the deposition of lead, or of SO_4'' in the deposition of silver, actions which are not explained by the theory of Blum and Rawdon (B., 1923, 1076). The observation that the polarisations at given current density in the deposition of the "frost-flower" and the fine-grained forms of tin are identical within the experimental error (1 millivolt) is, however, not necessarily in disagreement with that theory. H. J. T. Ellingham.

Influence of prolonged heating on resilience of some exhaust-valve metals. J. F. Saffy (Compt. rend., 1926, 183, 861—863).—The effect of prolonged heating at temperatures below the transition point has been studied for four steels (composition given), the samples being heated for periods up to 12 weeks in a furnace kept at 600°; two of the steels were also treated at 850°. The resilience of the samples was then determined, and by far the most satisfactory was an austenitic nickel–chrome steel, the final resilience of which was greater than the initial value. S. J. Gregg.

Electric annealing of magnetic materials for telephone apparatus. W. A. Timm (Trans. Amer. Soc. Steel Treat., 1926, **10**, 782—799).—Coal- and oil-fired annealing furnaces have been replaced by a recuperative electric furnace treating six tons of telephone parts daily. The furnace is of the car type, eight annealing pots weighing 3000 lb. when charged being loaded on each car. No packing material is used around the parts in the pots. The furnace has two tracks, the cars passing in opposite directions through the three furnace compartments. Heating elements are placed only in the central compartments, but there are elements immediately below the top plate of each car. A cold charge entering an end compartment is preheated by a hot charge in process of cooling down. A car stays approximately 6 hrs. in each compartment. The maximum power input is 158 kw. Temperature control is automatic. The maximum temperature difference between individual pots is never more than 16°. Tests show that the magnetic quality and uniformity of the material are improved. T. H. Burnham.

Patents.

Production of wrought iron. E. F. Blessing (E.P. 261,449, 17.7.25).—Pig iron is refined in a furnace provided with hollow trunnions which rest on supporting rollers and connect with a chimney-shaft. The contour of the refractory lining of the furnace is designed so as to form a series of hearths for the several stages of the process. At the commencement, when the metal is in a liquid state, the hearth has a large radius of curvature, so that a large area of surface of the metal is exposed to the oxidising gases in the furnace. As the furnace is tilted the curvature of the bed increases, and finally, the iron, beginning to "ball up," is crowded into a comparatively deep and narrow section producing a rounded form or ball. C. A. King.

Production of iron castings with a low carbon content. K. Emmel, and Thyssen & Co., A.-G. (E.P. 244,405, 11.6.25. Conv., 9.12.24).—For the production of castings having a low carbon content, the charge for the cupola consists of 50% or more of wrought iron or steel and the remainder pig iron, together with the usual additions. The coke required amounts to 9—13% of the charge, and the blast pressure to 400—800 mm. water gauge. The scrap metal is used preferably in the form of briquettes. C. A. King.

Manufacture of iron and steel alloys. W. B. Hamilton and T. A. Evans (E.P. 262,206, 24.9.25. Cf. J., 1922, 220 A; J., 1923, 1028 A; B., 1924, 338, 522).—High-percentage iron and steel alloys may be produced from scrap containing the alloying metal by melting together a mixture of rustless iron scrap, with or without mild steel scrap, and slag-forming materials consisting of a mixture of limestone, scale (iron oxide), fluorspar, and chromite, the quantities being sufficient to make a bath of the required mass and a slag having sufficient mass and heat to start and support the exothermic action when the reduction mixture is added. Sufficient reduction mixture (ferrosilicon and chromite) is now added to increase the chromium content to that required, and must contain a proportion of thermo-reducing agent 10—15% in excess of that theoretically required in order to reduce back again into the bath all the chromium which may have become oxidised into the slag during

melting. The carbon content of the bath during melting may be reduced by the addition of a suitable oxide such as mill-scale. The high-percentage steel alloy so produced is free from contamination from the carbon electrodes and from gases from the electric arc.

M. E. Nottage.

Process for treating blast-furnace slag and the like. A. and J. Crawford (E.P. 261,976, 26.3.26).—To reduce the tendency of blast-furnace slag to disintegrate on exposure to the atmosphere, acid steel slag (1 to 1½ cwt.), preferably finely ground, is added to blast-furnace slag (1 ton) after leaving the furnace and while still in a molten condition. C. A. King.

Treatment of magnesium or its alloys for casting and other operations. G. Michel (E.P. 261,528, 17.10.25).—A mixture of magnesium or its alloys with magnesium fluoride is heated and, while still in a semi-fluid condition, 0·05—0·3% of calcium is added in the form of small particles which will pass through a 5-mesh and be retained by a 50-mesh sieve. The addition of calcium lowers the m.p. of the metal or alloy, and causes vigorous agitation of the mass with subsequent separation of the magnesium fluoride, which rises to the surface carrying with it any chlorides and oxychlorides. The surface of the bath is then sprinkled with ammonium chloride, which reduces all the oxide impurities. Magnesium alloys are thus rendered perfectly stable, and are therefore adapted to all thermo-mechanical operations such as forging and rolling, and according to their composition possess hardness, elasticity, and ductility, and are capable of autogenous welding.

M. E. Nottage.

Solder for aluminium or its alloys and process for using same. A. J. Line (E.P. 262,192, 8.9.25).—The solder consists of a mixture of 3 pts. by wt. of pure silver and 12 pts. by wt. of aluminium heated together until the aluminium melts and the silver is absorbed by it; or filings of the metals may be mixed together and the solder used in this form. A nickel soldering bit is used without flux. M. E. Nottage.

Purification of gallium, indium, and similar metals, and their alloys. British Thomson-Houston Co., Ltd., Assees. of S. Boyer (E.P. 256,950, 30.7.26. Conv., 12.8.25).—A film of a halogen compound is first formed on the surface of the electrolytically-deposited metal either by treating it with dilute hydrochloric acid (1 : 1), fusing it under the acid, and then resolidifying it, or by heating the metal in a container in contact with a dry halogen gas, the air having been first withdrawn. The metal is then placed in a quartz container, and the halogen compound volatilised by heating it at about 500° in a vacuum. Occluded gas is removed by raising the temperature to about 1000°. The metal is cooled, reheated several times, and then transferred to a second receptacle connected with the container, in which it is sealed off from the latter and finally removed by distillation or otherwise. M. E. Nottage.

Manufacture of heavy-metal carbides and similar difficultly-melting compounds. Gewerkschaft Wallram (E.P. 242,951, 11.8.25. Conv., 14.11.24. Addn. to 239,499; B., 1925, 926).—Heavy-metal carbides, *e.g.*, tungsten monocarbide, are melted in a graphite container in an electric carbon tube furnace. The container forms part of a wedge-shaped block which extends outside the furnace for use as a handle, and in which a channel is cut to form a duct for the molten charge to run into a mould outside the furnace. Melting and carbon absorption are controlled by observation of a check block placed above the charge, and after a certain empirically determined time the charge is tapped in a condition of maximum fluidity. C. A. King.

Metallurgical process. C. L. Burdick, Assr. to Guggenheim Bros. (U.S.P. 1,606,343, 9.11.26. Appl., 7.6.24).—Volatile sulphur compounds of metals may be formed from metal-bearing material by subjecting the latter, at an elevated temperature, and in the presence of a sulphur component, to the action of a reducing gas in intimate contact with it. The volatile product is withdrawn and condensed. M. E. Nottage.

Protective coatings for metals. N. E. North (E.P. 261,502, 17.9.25).—Iron or steel articles may be "sherardised" by heating them in contact with, or immersed in, a finely-powdered mixture of zinc with 1—20% of cadmium, with or without the addition of sand and ground carbon. The heating is continued at a temperature well below that of the m.p. of zinc, the furnace being rotated or periodically tilted during the heating.

M. E. Nottage.

Protection of ferrous metals against corrosion. G. B. Ellis. From T. E. Murray (E.P. 261,809, 24.6.25).—Ferrous metal is coated, preferably electrolytically, with chromium and nickel, and the coated metal heated so as to cause the chromium to alloy with the iron and to form an adherent oxide coating. Successive coatings of chromium, iron, chromium, and nickel may be subsequently applied. J. S. G. Thomas.

Means for making gas-tight joints between metal and glass or quartz. Internat. General Electric Co., Inc., Assees. of Allgem. Elektricitäts-Ges. (E.P. 243,000, 12.11.25; Conv., 12.11.24. Addn. to E.P. 239,871).—The unglazed, slightly corrugated end of the glass or quartz is sprayed with metal, *e.g.*, copper or brass, and the layer is tinned. The metal cap is then attached, preferably with the interposition of a ring washer, by soldering a metal casing over the cap and tinned layer. A. Cousen.

Sealing metal to glass. N. V. Philips' Gloeilampen-Fabr. (E.P. 251,951, 1.4.26. Conv., 11.5.25).—An alloy suitable for making an air-tight seal between metal discs, plates, wires, etc., and glass, consists of approximately 25% Cr, up to 5% Al, remainder Fe.

A. Cousen.

Sealing molybdenum or similar refractory metals and fused quartz. H. Miller, Assr. to Hanovia Chem. and Manuf. Co. (U.S.P. 1,608,612, 30.11.26. Appl., 8.1.26).—Molybdenum and allied metals may be united with fused quartz by fitting the metal on to the quartz, treating it with certain salts of the alkali metals, heating the metal at its junction with the quartz until the latter becomes plastic, and then stressing or pressing the quartz until it flows and blends with the metal.

M. E. Nottage.

Alloys suitable for loading telephone and telegraph conductors. W. S. Smith and H. J. Garnett

(E.P. 261,060, 10.7.25).—Alloys having a high sp. resistance and high initial magnetic permeability are composed of 62—69% Ni, 11—19% Fe, 6—10% Cu, 5—9% Cr, with (or without) not more than 0·5% Mn as a deoxidiser. An alloy containing 65% Ni, 18% Fe, 10% Cu, 7% Cr, and 0·5% Mn has $\mu = 1200$, $\rho = 99$ microhms/c.c. F. G. Crosse.

Alloy [for electrical contacts]. H. M. Fredriksen (U.S.P. 1,605,432, 2.11.26. Appl., 2.8.23).—An alloy containing 87% Ag, 12% Cu, and 1% C. C. A. King.

Producing "non-sag" tungsten [filament] wire. P. McAllister, Assr. to Westinghouse Lamp Co. (U.S.P. 1,605,192, 2.11.26. Appl., 23.12.22).—Tungsten filaments which do not sag in use are obtained by flashing tungsten wire in the vapour of a reducing agent, such as phosphorus or arsenic, either before or after insertion in the electric lamp.
T. S. Wheeler.

Alloy steel. H. B. Kinnear, Assr. to Marion Steam Shovel Co. (U.S.P. 1,607,086, 16.11.26. Appl., 30.12.25). —See E.P. 260,835; B., 1927, 46.

Method for decarbonising highly carbonaceous ferrochromium. L. Friderich and W. Rodenhauser, Assrs. to Siemens & Halske A.-G. (U.S.P. 1,608,270, 23.11.26. Appl., 5.9.25. Conv., 26.6.24).—See F.P. 600,079; B., 1926, 412.

Process and apparatus for casting liquid molten metal. A. Kadow, Assr. to Vacuum Casting Co. (U.S.P. 1,606,236, 9.11.26. Appl., 15.3.26).—See E.P. 260,743; B., 1927, 47.

Treatment of oxidised ores or other oxidised compounds of copper and zinc. G. W. Edwards and H. T. Durant (U.S.P. 1,608,844, 30.11.26. Appl., 10.11.23. Conv., 20.11.22).—See E.P. 215,802; B., 1924, 601.

Magnetisable material [alloys]. E. Schürer, Assr. to Felten und Guilleaume Carlswerk A.-G. (U.S.P. 1,608,434, 23.11.26. Appl., 13.8.25. Conv., 30.8.24).—See E.P. 239,231; B., 1925, 925.

Puddling furnaces. E. F. Blessing (E.P. 261,812, 17.7.25).

Metallurgical apparatus [moulds etc.]. Soc. Anon. Métallurgique d'Aubrives et Villerupt (E.P. 261,269, 29.4.26. Conv., 3.4.26).

XI.—ELECTROTECHNICS.

High-frequency [induction] furnaces. J. Bronn (Z. Metallk., 1926, **18**, 333—338).—A discussion of the principles and efficiency of high-frequency inductive heating, with especial reference to the Ribaud furnace.
A. R. Powell.

Investigations relating to the pyrolusite [Leclanché] cell. K. Arndt, H. Walter, and E. Zender (Z. angew. Chem., 1926, **39**, 1426—1429).—The electrochemistry of the "dry" Leclanché cell is studied. The chemical changes occurring during discharge of the cell are shown to be represented by $Zn + 2NH_4Cl + 2MnO_2 = Zn(NH_3)_2Cl_2 + H_2O + Mn_2O_3$, and $Zn + H_2O + 2MnO_2 = Zn(OH)_2 + Mn_2O_3$. The carbon–manganese dioxide electrode must be considered as a true oxygen electrode, atmospheric oxygen taking part in the chemical changes involved during discharge of the cell.
J. S. G. Thomas.

Electric annealing of magnetic materials for telephone apparatus. Timm.—See X.

Patents.

Electrolyser for the production of hydrogen and oxygen. "Montecatini" Soc. Gen. per l'Ind. Mineraria ed Agricola, Assees. of G. Fauser (E.P. 242,635, 30.10.25. Conv., 7.11.24. Cf. U.S.P. 1,495,681; B., 1924, 633).—The electrodes employed are each enclosed in an asbestos canvas bag, and comprise two parallel plates spaced apart for circulation of an electrolyte and uniformly divided by vertical slots forming channels extending from their top to bottom edges. T-shaped spacing members are provided between adjacent vertical edges of the plates. J. S. G. Thomas.

Process and apparatus for electrodeposition. International Copperclad Co., Assees. of T. Robinson (E.P. 238,230, 4.8.25. Conv., 5.8.24).—Apparatus for electrodeposition applicable to the manufacture of roofing elements comprises an anode and cathode suspended side by side and carried together through an electrolyte. Thus films of lead and copper may be successively deposited on a cathode, the films stripped from the cathode, and non-metallic bases fixed to them.
J. S. G. Thomas.

Electrolytic apparatus. A. E. Knowles (E.P. 261,080, 11.8.25).—To maintain the temperature of the electrolyte, tubes or coils, through which hot or cold fluid may be circulated, are arranged adjacent to the ends of the electrodes and at right angles to their planes. J. S. G. Thomas.

Electrolytic apparatus. A. E. Knowles (E.P. 261,164, 19.10.25).—Water or other fluid, fed into one or more chambers in which the gas or gases evolved from the electrolytic cell bubble through the fluid, passes into a chamber containing a float operating a valve controlling the supply of fluid to the former chamber or chambers, and then flows to the cell in order to maintain the level of electrolyte therein. J. S. G. Thomas.

Manufacture of metal sheets by electrodeposition. A. Z. Pedersen (E.P. 261,592, 9.2.26).—A number of separable sheets of an anodically-corrosive metal, more especially nickel or cobalt, are deposited upon a cathode in an electrolytic bath, the direction of the current being reversed between the deposition of each sheet for a period of time which is short compared with that necessary for the deposition of a sheet. J. S. G. Thomas.

Manufacture of an electrolyte. A. Miller (U.S.P. 1,605,756, 2.11.26. Appl., 19.7.24).—An electrolyte is composed of litharge, lead sulphate, lead peroxide, and sulphuric acid, which has been subjected to the action of an electric current, and from which surplus acid has been removed. J. S. G. Thomas.

Diaphragms for primary or secondary cells, electrolytic cells, filters, and the like. H. Beckmann (E.P. 238,870, 6.8.25. Conv., 22.8.24).—A spongy rubber diaphragm having a large number of extremely minute pores is produced by coagulating latex or other suitable rubber dispersion by solutions of magnesium or

barium salts or other coagulating agents. If desirde the pores may be impregnated with a solution of wood-extract, phenol, etc. J. S. G. THOMAS.

Depolarising composition [for dry cells]. G. W. HEISE, Assr. to NATIONAL CARBON CO., INC. (U.S.P. 1,605,582, 2.11.26. Appl., 29.10.23).—A depolarising composition, for use in dry cells, is obtained by extracting diatomaceous earth with dilute sulphuric acid, grinding the purified earth with carbon, and incorporating the mixture with manganese dioxide. T. S. WHEELER.

Electro-deposition of metals. C. P. MADSEN, Assr. to MADSENELL CORP. (U.S.P. 1,608,706, 30.11.26. Appl., 26.4.19. Renewed 20.12.24).—See E.P. 142,432; B., 1921, 87.

Electrical deposition of organic materials [rubber]. S. E. SHEPPARD and L. W. EBERLIN, Assrs. to KODAK, LTD. (E.P. 253,085, 30.4.26. Conv., 8.6.25).—See U.S.P. 1,580,795; B., 1926, 639.

Thermo-electric element. O. HERMANN, Assr. to THERMO ELECTRIC BATTERY CO. (Reissue 16,453 of U.S.P. 1,572,117, 9.2.26. Appl., 4.5.25).—See B., 1926, 371.

Electrical treatment of gases and vapours (U.S.P. 1,601,771).—See II.

Refining petroleum [by electrical precipitation] (U.S.P. 1,604,424).—See II.

Protection of ferrous metals against corrosion (E.P. 261,809).—See X.

Alloys suitable for loading telephone and telegraph conductors (E.P. 261,060).—See X.

Producing "non-sag" tungsten [filament] wire (U.S.P. 1,605,192).—See X.

Alloy [for contacts] (U.S.P. 1,605,432).—See X.

XII.—FATS; OILS; WAXES.

Rate of molecular weight increase in the boiling of linseed and tung oils. J. S. LONG and G. WENTZ (Ind. Eng. Chem., 1926, **18**, 1245—1248).—Only moderate concordance is obtained when various solvents are used in the determination of mol. wts. of thickened linseed and tung oils by the cryoscopic method, and benzene is recommended as a regular solvent. For thickened oils with mol. wt. over 2000, it is advisable to extract with hot benzene in a Soxhlet apparatus and adjust the concentration of the extract to 1%, the standard concentration adopted. The rates of mol. wt. increase of linseed oil heated alone and with various additions were determined. The presence of linolenic monoglyceride or of linolenic acid increases the rate whilst free glycerol retards it. Retardation is also induced by thickening the oil in an atmosphere of steam. These experiments, and a further one in which linseed oil was thickened in an atmosphere of carbon dioxide and steady evolution of water, acraldehyde, etc. took place coincidently with uniform rise in mol. wt. and fall in iodine value, are proffered as evidence for the occurrence of condensation reactions. The removal of mucilage from linseed oil accelerated the increase of mol. wt. on heating. A significant fact in connexion with commercial oil boiling was the development of mucilage in a "non-breaking" oil by the use of an atmosphere of steam, since varying amounts of water, inadvertently introduced, may develop the "break," with subsequent adsorption of driers which are thus rendered ineffective. The addition of sulphur to linseed oil caused a rapid increase of mol. wt. while the oil was heating up, but a considerably retarded rate of increase on maintaining the oil at 293°. S. S. WOOLF.

Rate of molecular weight increase in the boiling of linseed oil. J. S. LONG and W. J. ARNER (Ind. Eng. Chem., 1926, **18**, 1252—1253).—The effects of various driers on the rate of increase of the mol. wt. of linseed oil on heating are tabulated. Preliminary attempts are made to couple the various maxima of such physical properties of oil films as elongation or ultimate tensile strength with the molecular state of the oil from which the films are derived. S. S. WOOLF.

Relationship during drying between the acid value of linseed oil and the concentration of cobalt acetate. W. L. EVANS, P. E. MARLING, and S. E. LOWER (Ind. Eng. Chem., 1926, **18**, 1229—1230).—The acid value of linseed oil films containing varying concentrations of cobalt acetate, drying under standard conditions, is an increasing function of the time. The acid value is also expressed as a function of the cobalt acetate concentration, graphically and by a series of exponential equations. S. S. WOOLF.

Rate of polymerisation of perilla oil. M. TOCH and T. T. LING (Ind. Eng. Chem., 1926, **18**, 1285—1287).—The polymerisation of perilla oil, examined at temperatures up to 305°, is shown to proceed with the evolution of volatile matter probably including acraldehyde. The original oil of d^{20} 0·9134, n^{25} 1·4812, acid number 3·10, and iodine value (Wijs) 201 gives at the end of 3 hrs. "bodied" oils having the characteristics d^{20} 0·9672 and 0·9722, n^{25} 1·4914 and 1·4923, acid number 13·62 and 12·22, and iodine value 141 and 138 according as the polymerisation is conducted by heat alone or by heat and "blowing," the latter oil being much darker in colour. Tables are given showing the values obtained for samples taken from a larger scale experiment in which the heating was continued for 8 hrs. It is demonstrated that the mean mol. wt. of the oil rises uniformly with the density, refractive index, and acid value, and inversely as the iodine value, between the values 753 at the start and 1650 after 8 hrs. E. HOLMES.

Fatty acids of whale oils. II. Fatty acids of long-headed finner (sei) whale oil. Y. TOYAMA (Chem. Umschau, 1926, **33**, 293—299, and J. Soc. Chem. Ind. Japan, 1926, **29**, 531—538)).—The oil under examination, of d_4^{30} 0·9106, d_4^{15} 0·9203, acid value 0·98, saponif. value 168·3, iodine value (Wijs) 121·3, n^{30} 1·4699, n^{20} 1·4736, and unsaponif. matter 2·29%, gave mixed fatty acids having the following characteristics: d_4^{30} 0·84, acid value 192·2, saponif. value 194·1, iodine value (Wijs) 125·9, n^{30} 1·4609, and ether-insoluble bromides 25·59%. By methods similar to those used in Pt. I (B., 1924, 1019) the fatty acids are separated and identified as follows. The saturated acids amount to 20% and those of the oleic series to not more than 75% of the total acids. The saturated acids consist largely of palmitic acid; myristic, stearic, and arachidic acids are present in

smaller amounts, whilst behenic acid and acids of fewer carbon atoms than 14 appear in very small amounts. Acids of the oleic series are zoomaric, oleic, an acid $C_{20}H_{38}O_2$, and cetoleic acid $C_{22}H_{42}O_2$. There is a possibility of the presence of tetradecenoic acid and of unsaturated acids having more than 22 carbon atoms in very small amount. The unsaturated acids with more than one double linking consist largely of those with 20 and 22 carbon chains, with smaller amounts of 18-carbon acids, including $C_{18}H_{28}O_2$ and $C_{18}H_{30}O_2$. $C_{20}H_{32}O_2$ occurs in the C_{20} acids, and among the C_{22} acids is $C_{22}H_{34}O_2$—probably clupanodonic acid—and smaller amounts of $C_{22}H_{36}O_2$.

E. Holmes.

Dibromides of oleic and elaidic acids and the purification of oleic acid. D. Holde and A. Gorgas (Z. angew. Chem., 1926, **39**, 1443—1446).—Crude oleic acid, iodine value (Hanus) 92·14, is treated with bromine, and the resulting product dissolved in light petroleum (b.p. 30—50°). By successive fractional crystallisations at − 21° and − 78° oleic acid dibromide is obtained as a solid, m.p. 28·5—29°. Elaidic acid, m.p. 43—44°, similarly treated yields the dibromide, m.p. 29—30°, the crystalline form of which is obviously different from that of its isomeride, mixed m.p. 20°. Oleic acid obtained by reduction of its dibromide distils at 200°/0·6 mm., has iodine value (Hanus) 89·96, and m.p. 13·5° after cooling at 0° for ¼ hr., 14·0° after 3 hrs. The results obtained confirm the findings of Nicolet (A., 1921, i, 390) that the addition and subsequent removal of bromine cause no *cis–trans* isomerisation in this series. E. Holmes.

Fatty acids of whale oils. IV. Fatty acids of Californian grey whale oil. Y. Toyama (J. Soc. Chem. Ind. Japan, 1926, **29**, 538—546, and Chem. Umschau, 1927, **34**, 19—25).—The fatty acids of Californian grey whale oil of d_4^{15} 0·9290, acid value 0·50, saponification value 191·0, iodine value 166·7, n_D^{20} 1·4788, unsaponifiable matter 1·55%, were examined. The mixed fatty acids contain about 10% of saturated acids and the unsaturated acids about 60% or less of acids of the oleic series. Amongst the saturated acids, palmitic acid preponderates, with myristic and stearic acids in smaller amount, and a trace of arachidic acid. The presence of a small amount of saturated acids having less than 14 carbon atoms is also indicated. The acids of the oleic series consist mainly of zoomaric and oleic acids. $C_{20}H_{38}O_2$ and $C_{22}H_{42}O_2$ are also present. A tetradecenoic acid, $C_{14}H_{26}O_2$, is present in a small amount. The acids more unsaturated than those of the oleic series consist chiefly of C_{20} and C_{22} acids, mainly $C_{20}H_{32}O_2$ and $C_{22}H_{34}O_2$, also $C_{20}H_{30}O_2$, $C_{24}H_{30}O_2$, and $C_{22}H_{36}O_2$. C_{16} and C_{18} acids ($C_{16}H_{26}O_2$, $C_{16}H_{24}O_2$, $C_{18}H_{28}O_2$, $C_{18}H_{30}O_2$) also occur in the acids giving ether-insoluble bromides.

K. Kashima.

Fatty oils as substitutes for ethyl alcohol in citrus flavours. H. A. Schuette and B. P. Domogalla (Ind. Eng. Chem., 1926, **18**, 1254—1257).—In a search for substitutes for ethyl alcohol in the preparation of orange and lemon flavours the following fatty oils have been examined: sweet almond, corn, cottonseed, neutral lard, olive, peanut, rape, sesamé, and soya-bean oils, all of minimum acidity. The solutions are analysed by a polariscopic method in a 100 mm. tube, the appropriate factors for lemon and orange oils being 1·7 and 2·7 respectively. Baking tests on flavours consisting of 5% by volume of the essential oils in the various fatty oils show them to be equal to, if not better than, the alcoholic extracts, but the lack of stability of such flavours militates against their general use. Acid numbers of these solutions show in 54 months an increase of 25—55% for the lemon flavours, and 19—100% for the orange flavours. E. Holmes.

Detergent action of soaps. R. M. Woodman (Chem. News, 1926, **133**, 339—341, 353—356).—Phenol, hydrogenated phenols, and cresylic acid are much more soluble in soap solutions than in pure water, and addition of these substances greatly increases the solvent power of soap solutions for paraffin oil, anthracene oil, coal-tar creosote, etc. Vegetable oils and oleic acid were found to be quite insoluble. Pickering (B., 1917, 462) has shown that the more concentrated a soap solution the greater is the solvent action per unit of soap. The present measurements show, however, that the influence of concentration is not of such importance when phenolic substances are added to the soap. The presence of phenols, hydrogenated phenols, or cresols renders soaps much more soluble, and more easily soluble, in water, yielding clear solutions. Further, besides possessing greater solvent and emulsifying powers for hydrocarbon oils, solution and emulsification are much more rapid, thus tending to increase detergent action.

J. S. Carter.

Apparatus for the determination of moisture [in soap] by distillation with xylene. Pritzker and Jungkunz.—See I.

Effects of ultra-violet light on paint vehicles. Stutz.—See XIII.

Function of oils and fats, and emulsions of oil and water in breadmaking with special reference to gluten formation and modification. Bennion.—See XIX.

Patents.

Refining and decolorising oils and fats. A. S. Quick (E.P. 261,440, 18.6.25).—Gases rich in oxygen are passed through the oil or fat to be refined, in the presence of water and a radioactive material, *e.g.*, monazite sand. The addition of very small quantities of alkaline substances and/or substances capable of absorbing pigments, *e.g.*, charcoal, fuller's earth, etc., is sometimes necessary. Temperature and duration of treatment depend on the nature and proposed use of the oil treated. (Cf. E.P. 161,813; B., 1921, 397.) S. S. Woolf.

Manufacture of butter fat products (U.S.P. 1,605,009).—See XIX.

Process of producing edible fat (U.S.P. 1,605,108).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Application of the statistical method in testing paints of durability. J. H. Calbeck (Ind. Eng. Chem., 1926, **18**, 1220—1222).—Results of exposures on the N. Dakota test fences (1915 and 1921) are statistically analysed, enabling the influence on durability of particular

variables to be clearly defined. The significance of the "volume ratio" (percentage by volume of the pigment phase in the dried paint film) is established, 28% being the probable minimum compatible with satisfactory durability, but the supposed existence of an optimum percentage of zinc in mixed lead-zinc paints is not confirmed. The extent of "chalking" after one year's exposure of a paint is not a reliable indication of its durability. S. S. WOOLF.

Accelerated weathering. H. A. NELSON and F. C. SCHMUTZ (Ind. Eng. Chem., 1926, **18**, 1222—1227).—The destructive agencies made use of in laboratory accelerated weathering cycles are correlated with their counterparts in the actual weathering process. From consideration of the effects of ultra-violet light, alone and in conjunction with heat, humidity, and oxygen- or ozone-enriched atmospheres, the solvent and erosive functions of water sprays, and the varying action of temperature changes according to the original elasticity of the film under test, a cycle is devised which successfully simulates the weather conditions prevailing in a particular locality (Palmerton, Pa., U.S.A.). S. S. WOOLF.

Cause and prevention of staining on white paint. H. T. MORGAN and J. H. CALBECK (Ind. Eng. Chem., 1926, **18**, 1227—1228).—The staining of white paints in contact with iron or copper is shown to be due to the presence of zinc oxide, which, unlike other white pigments, precipitates colloidal metal hydroxides from solution. S. S. WOOLF.

Effects of ultra-violet light on paint vehicles. G. F. A. STUTZ (Ind. Eng. Chem., 1926, **18**, 1235—1238).—The exposure of linseed, perilla, poppy seed, soyabean, and castor oils, in mass, to the action of ultra-violet light at 50° (with the passage of a slow stream of air, oxygen, or nitrogen through the oil) produced similar results to the application of heat or air-blowing, *i.e.*, decrease in iodine value, and increase in viscosity, mol. wt., refractive index, and acid value. The degree to which ultra-violet light (over the wave-length interval from 3655—2300Å) is absorbed by oils was observed by measuring the light transmitted through a cell containing the oil under test, using an ultra-violet spectrophotometer. This was carried out for the various oils, before and after treatment with ultra-violet light, as well as for several commercial samples of treated and bodied linseed oils, and various fatty acids etc. When the degree of absorption shown by the oil is expressed by the relationship $I = I_0.10^{-Kt}$, where I = intensity of transmitted light, I_0 = intensity of incident light, t = film thickness, and K = absorption coefficient, K is found to increase with mol. wt. and viscosity in all cases. Exposure to light has, therefore, a cumulative effect by making the oil more absorbent for ultra-violet rays. S. S. WOOLF.

Developments in controlling tung oil varnishes. W. W. BAUER (Ind. Eng. Chem., 1926, **18**, 1249—1251).—A *résumé* of current views on the solidification in sunlight, gelatinisation under heat, and frosting or "crystallising" of tung oil is given. The "burnt coal gas" test for producing frosting of tung oil varnish films is criticised, and it is established that the presence of oxygen is essential for "crystallisation," whereas both the formation of a solid modification and the production of frosted films have been achieved in the absence of light (contrary to previous statements). The rate and extent of "crystallisation" are increased by factors which permit an increased rate of oxygen absorption, oxides of nitrogen being found to act catalytically in this sense. A test method is described, according to which tung oil varnish films remain for 1 hr. at ordinary temperatures in a uniformly distributed atmosphere supplied by bubbling air through concentrated nitric acid. Varnishes that do not frost under these conditions may be expected to dry glossy under all normal conditions. The tendency to "crystallise" in varnishes is marked by the development of a surface gel, which may re-dissolve. S. S. WOOLF.

Analysis of lacquers. B. J. OAKES (Ind. Eng. Chem., 1926, **18**, 1232—1233).—In a general discussion of lacquer analysis the importance of preliminary physical tests is stressed, such phenomena as "sweating" or discoloration under heat, brittleness on bending, etc. affording useful guidance in the subsequent stages. For actual analysis, a recommended procedure is as follows:—100 g. of lacquer are stirred into about 800 c.c. of toluene. The pigment stays with the gelled nitrocellulose, and by repeated extractions with benzene the other ingredients are removed practically entirely and further examined. The nitrocellulose is separated from the pigment by extraction with acetone and centrifuging. Synthesis as a confirmation of analytical results is necessary. S. S. WOOLF.

Physical constants and individual properties of cellulose lacquer solvents, thinners, and plasticisers. C. P. VAN HOEK (Farben-Ztg., 1926, **32**, 624—628).—The following particulars of over 200 organic substances are tabulated:—formula, b.p./760 mm., m.p., d^{15}, n^{20}_{D}, α_D, inflammability, odour, physiological action, solubility, and solvent action. S. S. WOOLF.

Interpretation of stress-strain curves on lacquer and lacquer components. W. A. McKIM (Ind. Eng. Chem., 1926, **18**, 1239—1242).—From consideration of the stress-strain curves of experimental lacquer films of various ages, subjected to variations in temperature, humidity, and light, the influence of specific constituents, *e.g.*, plasticisers, high-boiling solvents, resins, pigments, on strength and elasticity is discussed. S. S. WOOLF.

Varnish and stoving lacquers for preserve cans. H. SERGER (Farben-Ztg., 1926, **32**, 685—686).—The requirements (legal and general) of, and methods of testing, protective coatings for the inside of preserve tins are described. Temperature of stoving is shown to be of importance. An electrochemical explanation is offered of the observed fact that corrosion is more severe in cases of damage to the stoved lacquer film than if no varnish had been applied. S. S. WOOLF.

Determination of metallic lead in pigments. MCINTOSH.—See X.

Boiling of linseed oil and tung oil. LONG and WENTZ.—See XII.

Boiling of linseed oil. LONG and ARNER.—See XII.

Drying of linseed oil. Evans and others.—See XII.

Polymerisation of perilla oil. Toch and Ling.—See XII.

Patents.

Preparation of titanium compounds [for pigments]. P. Spence & Sons Ltd., W. B. Llewellyn, and S. F. W. Crundall (E.P. 261,051, 8.5.25).—Precipitated hydrated basic titanic phosphate is obtained in a filterable and washable form by slowly adding a solution of sodium phosphate or phosphoric acid, while stirring, to a solution of titanium sulphate or chloride containing less than 10% TiO_2, and free hydrochloric acid, at 80—100°, in the presence of finely divided barium sulphate. H. Royal-Dawson.

Manufacture of a brown pigment containing titanium sludge. Deutsche Gasglühlicht-Auer-Ges.m.b.H. (E.P. 257,229, 22.6.26. Conv., 22.8.25).—The sludgy residues obtained by treating titaniferous ores with sulphuric acid are neutralised, washed, dried, calcined, and ground. The final stage of neutralisation is effected by the use of zinc oxide, strong alkalis being used up to this point. The colour of the finished pigment is determined by the temperature of calcination. S. S. Woolf.

Producing a titanium [oxide] pigment. R. H. Monk, Assr. to J. Irwin (U.S.P. 1.605,851, 2.11.26. Appl., 21.3.25).—Precipitated titanium hydroxide, free from iron, mixed with a filler, such as barium sulphate, and sawdust or other carbonaceous material (8%), is calcined at about 800°, when a pure white titanium oxide pigment is obtained. T. S. Wheeler.

[Cellulose] varnish. E. Lavadoux (E.P. 261,589, 5.2.26).—To a cellulose varnish comprising nitrocellulose, castor oil, amyl or ethyl acetate, and benzene or benzine, is added "Oriental essence," a suspension of fish scales in aqueous ammonia. The mixture yields a flexible, resistant, glossy, and permanent film. S. S. Woolf.

Lacquer for shoe heels etc. F. G. Edbrook (U.S.P. 1,607,516, 16.11.26. Appl., 11.7.23. Conv., 26.7.22).—A lacquer containing equal proportions of cellulose acetate and dichlorohydrin, and a solvent consisting of acetone, together with benzyl alcohol, industrial alcohol, and benzene. H. Royal-Dawson.

Manufacture of resinous compositions. Metropolitan-Vickers Electrical Co. Ltd., Assees. of H. C. P. Weber (E.P. 253,519, 7.6.26. Conv., 10.6.25).—A phenol–furfuraldehyde resin is mixed with a "glyptal" (glyceryl phthalate) resin (both being in either the A- or B-stage of condensation) with or without fillers or the use of a solvent. The composite resin is "cured" to any required stage, and retains the good insulating and resistant properties of the "glyptal" resin, together with the ease of moulding of phenol–furfuraldehyde resins. The improved rate of conversion to the C-stage suggests a mutual condensing action of the two types of resin. S. S. Woolf.

Synthetic resins. Chem. Fabr. Dr. K. Albert G.m.b.H., A. Amann, and E. Fonrobert (E.P. 261,522, 12.10.25).—The intermediate condensation product of phenols and aliphatic, aromatic, or mixed ketones is treated at a temperature not above 80° with an aldehyde in an alkaline medium. The resin subsequently precipitated with acid may be converted into an infusible, insoluble product by drying or heating. S. S. Woolf.

Hardening fusible phenolic resins. T. S. Huxham, Assr. to American Insulator Corp. (U.S.P. 1,606,943, 16.11.26. Appl., 27.3.24; cf. Mains and Phillips, B., 1921, 357 a).—Cold-moulding compositions suitable for the manufacture of insulators etc. are prepared by dissolving a fusible phenol–aldehyde resin (100 pts.) in a suitable high-boiling hydrocarbon solvent, and adding up to 90 pts. of the resinous product formed by the interaction of equimolecular proportions of furfuraldehyde and aniline, or other aromatic amine, and suitable amounts of fillers and pigments. The mixture is hardened when required by being heated at 150° for several hours. T. S. Wheeler.

Condensation product of acrolein and process of producing the same. C. Moureu and C. Dufraisse (U.S.P. 1,607,293, 16.11.26. Appl., 29.3.22. Conv., 31.3.19).—See E.P. 141,058; B., 1921, 858.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Patents.

Production of rubber isomer. H. L. Fisher, Assr. to B. F. Goodrich Co. (U.S.P. 1,605,180, 2.11.26. Appl., 26.12.24. Conv., 24.9.24).—See E.P. 249,172; B., 1926, 453).

Caoutchouc composition. A. Biddle, Assr. to United Products Corp. of America (Reissues 16,476—7, 16.11.26, of U.S.P. 1,437,487, 5.12.22).—See B., 1923, 107 a.

Manufacture of a rubberised fibre composition (U.S.P. 1,606,595).—See V.

Production of lampblack (U.S.P. 1,606,380). See II.

[Rubber] diaphragm for electrolytic cells etc. (E.P. 238,870).—See XI.

XV.—LEATHER; GLUE.

Maximum reactivity of the hide protein in its isoelectric zone. K. H. Gustavson (J. Amer. Chem. Soc., 1926, 48, 2963—2971).—This paper deals with a new type of protein reactions, which cannot be explained on the basis of a salt formation by means of primary valency. The salt studied was *cis*-sodium dioxalatodiaquochromiate $Na[(H_2O)_2Cr(C_2O_4)_2]$ (cf. Werner, A., 1915, i, 375). Solutions of this salt were adjusted by addition of *N*-sodium hydroxide so that four series of solutions, containing 5·0, 10·0, 24·0, and 55·0 g. of Cr_2O_3/litre, were obtained, each over a range of p_H 1—8. These solutions were aged for more than six months. Weighed amounts of American standard hide powder were shaken with measured volumes of these solutions for 48 hrs., the solutions were filtered, and the p_H values of the filtrates determined electrometically. The residues were analysed for chromium and protein. The fixation of the anionic chrome complexes from the oxalatochromiate was found to take place on both sides of the isoelectric point of the hide protein, with a pronounced

maximum in the region of that point. Since, at the iso-electric point, the formation of protein salts of primary valency nature is, as a rule, not possible, the production of molecular compounds by means of secondary valency forces from the basic protein groups to the central atom in the anion complex appears to be the most reasonable explanation of these results. Experiments with the oxalatochromiate solutions and de-aminised hide powder indicated that the chrome fixation reaction was practically independent of the extent of ionisation of the hide protein, since only a slight shift of the maximum zone was observed, and that the basic protein groups were chiefly involved in the anionic chrome fixation, a retardation of chrome fixation being obtained. This appears to indicate that in the fixation of cationic chromium the acidic protein groups are concerned (cf., Thomas and Kelly, B., 1926, 600). A number of facts are adduced to support this view of the dual nature of chromium fixation. For example, cationic chrome-tanned hide powder gives, upon treatment with solutions of oxalatochromiate, the same additional chrome fixation as a regular hide powder, but does not adsorb chromium from a cationic chromium salt. F. G. Willson.

Adsorption of acids by hide in relation to swelling. II. P. N. Pavlov and G. G. Timochin (Kolloid Z., 1926, **40**, 129—135. Cf. B., 1926, 958).—The hide was immersed in solutions of succinic or acetic acid for 24 hrs., and the initial and final concentrations of acid were determined by titration with phenolphthalein as indicator. From the apparent adsorption curves the true adsorption isotherms can be deduced. The adsorption coefficients of succinic and acetic acids are independent of the volume of the solution and mass of the adsorbent, and a partition of the acid between the aqueous and collagen phases evidently occurs.

E. S. Hedges.

Influence of hydrogen-ion concentration and of neutral salts on the degree of aldehyde tanning. O. Gerngross and R. Gorges (Z. angew. Chem., 1926, **39**, 1432—1435).—Hide powder is tanned for 5 hrs. in 0·95% formaldehyde solution of p_H range 3 to 12, and the degree of tanning of the resulting leathers determined as the "water-stability" number $= WB = 100\,(a - a')/a$, where a is the amount of hide substance originally present in the test sample, and a' the hide substance lost by extraction of the resulting leather with hot water under standard conditions. The method gives results reproducible to $\pm$ 1 unit. Tables of results and a graph demonstrate the following points: (*a*) The tanning, which is scarcely perceptible at p_H 3, rises slowly and uniformly to p_H 6·3; (*b*) between p_H 6 and 7 there is a relatively enormous rise in the amount of tanning; (*c*) between p_H 8 and 12 the degree of tanning is constant at the high value, $WB = 78$. Common salt solution 4/5 saturated and $0{\cdot}9N$- to $0{\cdot}09N$-potassium thiocyanate cause a sensible decrease in the degree of formaldehyde tanning of lamb skin, whilst the common salt itself in solution of p_H 4·5 has no appreciable effect. Saturated salt solution of p_H 2·5 to 3·6 suppresses all tanning, whereas a strongly alkaline solution, p_H 8·6 to 11·2, gives a beautiful leather of $WB = 87$. When lamb skin is tanned with formaldehyde solution to which has been added egg yolk the resulting leather is improved both in appearance and pliability.

E. Holmes.

Quantitative determination of gelatin. E. Lenk (Biochem. Z., 1926, **177**, 434—448).—The determination of gelatin in commercial samples and in glue cannot be effected by the viscosity method, since the latter is influenced by several factors, *e.g.*, p_H, ageing, salt content, homogeneity, etc. A new method is described, the percentage concentration of gelatin (y) being given by the relation $y = -0{\cdot}55x + 1{\cdot}8$, where x = logarithm of the gelatinisation time in min. The maximal error of the method is 2%, with times of gelatinisation ranging from 10 to 60 min. Commercial gelatin contains 70—100%, and glue 10—40% of gelatin. The curve showing the relationship between the time of gelatinisation and p_H has a sharp turning point between p_H 4·6 and 5·9; the freer the material from ash, the nearer is this point of inflexion to p_H 4·6. Between p_H 7·0 and 7·5 the curve becomes practically parallel with the abscissæ. Determinations of gelatin by this method are therefore carried out at this p_H (*i.e.*, after neutralisation to litmus).

P. W. Clutterbuck.

Patent.

Process of removing hair from hides or skins. H. C. Ross and H. C. Marris, Assrs. to W. Walker & Sons, Ltd. (U.S.P. 1,605,988, 9.11.26. Appl., 13.11.24). —See E.P. 241,666; B., 1926, 23.

XVI.—AGRICULTURE.

Studies on contact insecticides. I. Introduction and methods. II. Toxicity of *Tephrosia Vogelii*, Hook, to *Aphis rumicis*, L. III. Insecticidal action of chloro-, nitro-, and hydroxy-derivatives of benzene and naphthalene. IV. Toxicity of certain plants and plant products to *Aphis rumicis*, L. F. Tattersfield, C. T. Gimingham, and H. M. Morris (Ann. Applied Biol., 1925, **12**, 61—65, 66—76, 218—262; 1926, **13**, 424—445).—I. Methods adopted to obtain quantitative data with regard to the insecticidal properties of a number of groups of synthetic compounds and natural plant products are described. Adults of *Aphis rumicis* (black bean aphis) and eggs of *Selenia tetralunaria* (purple thorn moth) were used as test subjects. The insects were sprayed under controlled conditions, and afterwards kept under observation, counts being made of the numbers affected by the treatments. By testing compounds at a series of concentrations, the results can be graphed, plotting the concentrations against the percentages killed. II. Aqueous and alcoholic extracts of the leaves and seeds of the leguminous plant *Tephrosia Vogelii*, which occurs in many parts of the tropics, and in some districts is used as a fish poison, were highly toxic to *A. rumicis*. Aqueous extracts at concentrations equivalent to 1% or more of the leaves and seeds, and emulsions containing 0·1% or more of the dried alcoholic extract, were completely toxic. Extracts of the stems were less poisonous. III. The toxicity of a number of chloro-, nitro-, and hydroxy-derivatives of benzene and naphthalene to *A. rumicis* and eggs of *S. tetralunaria* was investigated. The introduction of chlorine or of a nitro-group into the benzene ring increases toxicity, and

a second nitro-group still further accentuates it. The following order of toxicity was noted: benzene < toluene < xylene < chlorobenzene < *p*-dichlorobenzene < *o*-dichlorobenzene < trichlorobenzene < nitrobenzene < *m*-dinitrobenzene. The nitro-phenols and -cresols are all more toxic than the parent substances. The dinitro-compounds are more toxic than the mono- or trinitro-compounds. The order of toxicity of the phenols was: phenol < *o*-nitrophenol < *m*- and *p*-nitrophenol < 2 : 4-dinitrophenol > trinitrophenol; and the same order applies to the cresols and their derivatives. With few exceptions, the relative toxicities to the aphides and to the eggs were approximately in the same order. α-Chloronaphthalene was the most toxic naphthalene derivative tested. 2 : 4-Dinitrophenol and 3 : 5-dinitro-*o*-cresol showed specially powerful insecticidal properties; the latter was completely toxic to the eggs at concentrations between 0·2 and 0·1%. It is suggested that compounds of this type, although injurious to foliage, may have practical value as winter spray fluids for fruit trees in dormant condition. (Cf. succeeding abstract.) IV. The toxicity to *A. rumicis* of extracts of a number of plants, including some tropical fish poisons, was studied. Alcoholic extracts of the roots and stems of white haiari and the stems of black haiari (species of *Lonchocarpus* from British Guiana), the roots of *Tephrosia toxicaria*, and the leaves of *T. Vogelii* all possessed high insecticidal properties. Certain substances isolated from these plants were also tested. The most toxic derivative obtained from the haiaris was shown to be identical with tubatoxin, the crystalline poison found in *Derris elliptica*. Tubatoxin was more toxic to *A. rumicis* than nicotine. Of a number of alkaloids tested, cytisine, lobeline, and eserine were rather less toxic than nicotine. None of the other alkaloids tried approached nicotine in toxicity.

C. T. GIMINGHAM.

Toxicity of 3 : 5-dinitro-*o*-cresol and other compounds to insect eggs, under laboratory and field conditions. C. T. GIMINGHAM, A. M. MASSEE, and F. TATTERSFIELD (Ann. Applied Biol., 1926, **13**, 446—465. Cf. preceding abstract).—Further laboratory experiments on the toxicity of 3 : 5-dinitro-*o*-cresol and its sodium salt to eggs of the moth *Selenia tetralunaria* are reported. The results confirmed the high toxicity of these compounds. The sodium salt was shown to be slightly less toxic than the dinitrocresol itself. Spray fluids containing 0·25 and 0·15% of dinitrocresol or the sodium salt were tested under field conditions, and showed a high efficiency against the eggs of the hop-damson aphis on plum trees. Counts of eggs on selected shoots on sprayed and control trees, before and after spraying, gave a reliable numerical measure of the effect of the treatments. No injury to the trees, due to the use of these spray fluids, was observed.

C. T. GIMINGHAM.

Determination of phosphoric acid in soils. D. J. HISSINK and M. DEKKER (Rijkslandbouwpraefstation, Groningen, 21.11.24; Bied. Zentr., 1926, **55**, 436—437). —For the determination of acid-soluble phosphate, 12·5 g. of air-dried and finely powdered soil are gently heated for 5 hrs. in a platinum crucible, with occasional stirring. The soil is then transferred to a flask and heated with 150 c.c. of 12·5% nitric acid on a glycerin bath (150°) for 75 min. After cooling, 5 c.c. of concentrated sulphuric acid are added, and the mixture is cooled and diluted to 250 c.c. and filtered. 50 c.c. of the filtrate are treated with 50 c.c. of sulphate-molybdate reagent (Lorenz), and, after 24 hrs., filtered (Neubauer–Tiegel method), and the precipitate is washed with ammonium nitrate solution and acetone, and dried for 30 min. *in vacuo*. Citric-soluble phosphate is determined by shaking 55 g. of air-dried, finely powdered soil with 500 c.c. of 2% citric acid solution for 48 hrs. at room temperature. 200 c.c. of the filtered extract (100 c.c. only if soil is rich in chalk) are evaporated to dryness with 50 c.c. of concentrated nitric acid. The residue is dissolved in a sufficiency of hot water containing 10 c.c. of 10% nitric acid, filtered, evaporated to about 15 c.c., treated with 35 c.c. of nitric–sulphuric acid solution and 50 c.c. of sulphate-molybdate solution, according to the Lorenz method, by which the analysis is completed.

A. G. POLLARD.

Action of lime on clay soils. D. J. HISSINK (Landbouwkundig Tijds., 1925, **37**, 442—444; Bied. Zentr., 1926, **55**, 484—486).—From determinations of the amounts of calcium carbonate and exchangeable calcium in limed soils, the author has calculated the percentage efficiency of dressings of lime on three different soils to be 80%, 3%, and 44% respectively. The differences are attributable partly to the character of the soils and partly to the mode of application of the dressings.

G. W. ROBINSON.

Action of lime on fen soils. D. J. HISSINK (Landbouwk. Tijds., 1926, **38**, 448—449; Bied. Zentr., 1926, **55**, 486—488. Cf. preceding abstract).—Data are given to show the fate of calcareous dressings applied to fen soil. The proportion of calcium oxide applied appearing finally in the form of "humus lime" was 87·5%, 95·1%, and 49·7% respectively in three cases, the last two being in different parts of the same field.

G. W. ROBINSON.

Action of lime on a Roodoorn soil. D. J. HISSINK (Landbouwk. Tijds., 1925, **37**, [45]; Bied. Zentr., 1926, **55**, 447—8).—The soil was a stiff clay with high humus content. The effect of liming on the saturation capacity, replaceable calcium, and p_H value of this soil is recorded. Liming was found to increase the availability of phosphates for plants.

A. G. POLLARD.

Determination of easily soluble soil phosphates. M. GERLACH and O. NOLTE (Mitt. Deut. Landw.-Ges., 1926, 10; Bied. Zentr., 1926, **55**, 448—450).—Comparison is made of results of phosphate determinations by a number of existing methods. Generally speaking, crop increases following the application of phosphatic manures, decreased with increasing amounts of citric-soluble phosphate in the original soils. Exceptions to this were frequent, however, and it was judged unsafe to use the method for evaluating fertiliser requirements. By employing the Neubauer method, it was observed that soils having a high "root-soluble" phosphate content were usually those benefiting least by treatment with phosphate fertilisers. No definite relationships were apparent, and exceptional cases were numerous. In the majority of soils examined, low citric-soluble phosphate contents were coincident with low "root-soluble" phosphate content. It is considered that field

trials alone offer a suitable basis for the determination of the phosphate deficiency of soils. A. G. POLLARD.

Phosphate solubility and the growth of *Azotobacter*. H. NIKLAS, K. SCHARRER, and A. STROBEL (Landw. Jahrb., 1926, **63**, 387—410; Bied. Zentr., 1926, **55**, 488—490).—The authors have studied the development of *Azotobacter* in cultures containing increasing amounts of various phosphates. The utilisation of the different phosphates as shown by the magnitude of the optimal additions can be correlated with their chemical solubility in different solvents.

G. W. ROBINSON.

Determination of the manurial requirements of soils. M. GERLACH (Landw. Jahrb., 1926, **63**, 339—368; Bied. Zentr., 1926, **55**, 481—484).—A critical review of the present position of the problem of determining the manurial requirements. G. W. ROBINSON.

Further manurial experiments with superphosphate, basic slag, Rhenania phosphate, and dicalcium phosphate on four different types of soil. H. NIKLAS, A. STROBEL, and K. SCHARRER (Landw. Jahrb., 1926, **63**, 607—625; Bied. Zentr., 1926, **55**, 493—495).—In field experiments on two neutral, one alkaline, and one slightly acid soil, respectively, over two seasons, superphosphate and Rhenania phosphate proved the most efficient phosphatic fertilisers. Dicalcium phosphate was less, and basic slag least effective. The high availability of the Rhenania phosphate may be correlated with its physical condition.

G. W. ROBINSON.

Adsorption capacity of soils. D. J. HISSINK (Chem. Weekblad, 1926, **23**, 511—515).—The view of adsorption as an exchange of cations between the solution and the soil, and the significance of the replacement of calcium and magnesium in the soil by sodium and potassium from the solution, are discussed. The following values for soil analysis are proposed:—(1) The milligram-equivalent of replaceable base in 100 g. of clay (*S* clay) and in 100 g. of humus (*S* humus); (2) saturation level of the soil, *V*, *i.e.*, ratio of replaceable bases to the total quantity the soil can bind, *T*; thus $V = 100\ S/T$; (3) lime factor (*Kf*), the quantity of lime which the humus must adsorb to give a neutral reaction ($p_H = 7$), and the relations between them considered. From the values of *T*, the average equivalent weights of clay and humus, respectively, are given as 1225 and 175. The relation between the amount of lime necessary to flocculate a soil suspension, the content of clay and humus, and the saturation level *V* is discussed. S. I. LEVY.

What happens to the lime when soil is limed? D. J. HISSINK (Proc. 2nd Comm. Int. Soc. Soil Sci., 1926, A, 174—197; Chem. Abstr., 1926, **20**, 3204).—The following data were obtained for experimental fields: p_H, *K* (humus) = g. humus — lime per 100 g. of humus, *K* (clay), *V* = degree of saturation, and the lime remaining as calcium carbonate in the soil. Four new values are employed: (1) Actual adsorption, *i.e.*, g. of lime adsorbed by the clay–humus substance per 100 g. of soil. (2) Potential adsorption, *i.e.*, g. of lime that the clay–humus substance per 100 g. of soil can take up under the prevailing climatic conditions, calculated (for Dutch conditions) on the assumption that the highest values of *K* are 5·2 (humus) and 1·1 (clay). (3) Coefficient of utilisation of the lime applied, *i.e.*, the actual adsorption as a percentage of the potential adsorption. (4) Liming ratio, *i.e.*, the ratio of g. of lime applied per 100 g. of soil to the potential adsorption. The largest possible actual adsorption in the shortest possible time with the smallest possible liming ratio is desirable. The actual adsorption will be the greater the finer-grained is the lime and the more thoroughly it is mixed with the soil immediately after liming. Soils with low *K* and *V* values adsorbed practically all the lime as clay–humus lime within about 1 yr., little remaining as calcium carbonate. The *K* and *V* values are predominant factors in determining the actual adsorption.

A. A. ELDRIDGE.

Relation between the p_H value, degree of saturation, and humus of some humus soils. Equivalent weight of the humus substance. D. J. HISSINK (Proc. 2nd Comm. Intern. Soc. Soil Sci., 1926, A, 198).—For 15 soils with humus content 6·7—50%, the p_H varies from 3·3 to 7·0, the degree of saturation (*V*) from 0·09 to 36·0, and the *S* (humus) from 6 to 214. Average values at p_H 7 were $V = 35{\cdot}8$; *S* (humus) = 206. The average equivalent weight of the humus substance is 176. A. A. ELDRIDGE.

Systematic methods for the recognition of plant extracts. GRIMME.—See XX.

PATENTS.

Process for preparing soil. A. WIDDIS (U.S.P. 1,608,917, 30.11.26. Appl., 17.12.24).—A layer of an emulsion of bentonite is placed at the bottom of the soil, previously loosened to a point below the usual depth of the plough. H. ROYAL-DAWSON.

Manufacturing colloidal phosphate fertilisers. W. DE HAËN (U.S.P. 1,607,666, 23.11.26. Appl., 20.1.25. Conv., 16.7.21).—Phosphatic material is treated with humus colloids at a low temperature and with moderate grinding in the presence of water.

H. ROYAL-DAWSON.

Ammonium salt [sulphate for fertiliser]. E. W. HARVEY (U.S.P. 1,608,738, 30.11.26. Appl., 16.8.22. Renewed 5.10.25).—The condition of commercial ammonium sulphate is improved by the addition of a mixture of a basic calcium compound and a semihydrated calcium sulphate. H. ROYAL-DAWSON.

Methods of making plant foods. H. F. BLACKWELL (U.S.P. 1,606,015, 9.11.26. Appl., 21.4.22).—A basic plant food element unavailable to plants is added to vegetable matter and decomposed by means of bacteria forming humic acid, which combines with the basic element forming a water-soluble humate which is recovered.

H. ROYAL-DAWSON.

Dressing seeds. FARBWERKE VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 241,568, 14.10.25. Conv., 15.10.24).—The emission of dust caused by using dry fungicide composition in dressing seeds is avoided by the addition of 3% of paraffin oil to such composition consisting of 30 pts. of copper carbonate, 30 pts. of a resin sulphonate, 5 pts. of phenylarsinic acid, and 32 pts. of kieselguhr. F. G. CROSSE.

Controlling the temperature of heated air in drying and like operations [for crops etc.] (E.P. 261,446).—Sec I.

Composition for exterminating weeds (E.P. 261,241).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Apparatus for the determination of moisture [in sugar] by distillation with xylene. PRITZKER and JUNGKUNZ.—See I.

Acid in potato flour and syrup. TRYLLER.—See XIX.

PATENTS.

Production of a cannery syrup for the canning and preserving of fruits and the products thereof. E. S. COCHRAN, Assr. to J. T. MCCROSSON (U.S.P. 1,606,827, 16.11.26. Appl., 19.8.24).—Without preliminary liming, the raw juice from the sugar cane or beetroot is strained through plate strainers and forced at a pressure of 20—30 lb./sq. in. through a closed heater at 104—110°. While under pressure it is filtered and further refined by the addition of 0·5% of porous infusorial earth and 1% of bone black, followed by a second filtration. The clear syrup, which contains the bulk of the sucrose and of the reducing sugars of the raw juice, is then concentrated. F. R. ENNOS.

Mixing methods and apparatus [for liming sugar juices] (E.P. 247,541).—See I.

Filtering device [for sugar solutions] (E.P. 261,204).—See I.

XVIII.—FERMENTATION INDUSTRIES.

Wort composition. H. L. HIND and N. C. BEETLESTONE (J. Inst. Brew., 1926, **32**, 540—551).—The increase in extract gained by acid addition is considerable when an alkaline mashing liquor is used. A greater extract results from the addition of vinegar than from sulphuric acid, but diastatic action is favoured more by the sulphuric acid and a greater proportion of reducing sugars is produced than when vinegar is added. In general, an unusually high proportion of buffer substances is undesirable, but the converse must not be pushed to extremes. Excess of buffers in the wort acting between p_H 5·5 and 4·0, by tending to inhibit the increase of acidity during fermentation, favours an unstable beer. Admixture of raw grain with malt containing excessive nitrogen increases the stability of the beer by producing a wort containing less buffer substances than an all-malt wort, and consequently does not hinder to the same extent the increasing production of acidity during fermentation. As sparging continues and as the worts become weaker the concentration of buffer substances decreases, whereas the relative proportion of buffers as compared to fermentable extract from the goods increases and a point may be reached when the disadvantages from excessive buffering may overbalance any advantage realised by further extraction of fermentable matter. C. RANKEN.

Yeast maltase. V. J. JSAIEV (J. Inst. Brew., 1926, **32**, 552—573).—Maltase is most active at a p_H of 6·1—6·7, the optimum p_H being independent of the temperature. The optimum temperature for its activity is 30—35°, and the temperature coefficient is 1·26. Even under aseptic conditions, its activity is greatly reduced by alcohol, acetone, and water, but is preserved better if kept in sterile solution in concentrated glycerol. Provided the period of action is short, the activity of maltase inhibited by contact with acids or alkalis may be restored. A more prolonged action of the acids or alkalis is followed by the destruction of the maltase. With low concentrations of maltase, the velocity of the hydrolysis of maltose is directly proportional to its amount, but proportional to the square root of the amount at higher concentrations. Dextrose and lævulose retard hydrolysis, whereas galactose has no effect. The mechanism of the reaction cannot be expressed by means of a single mathematical equation. Although maltose and dextrose form readily dissociated compounds with maltase, the affinity of the latter to maltose is much weaker than to dextrose. The enzymic hydrolysis of maltose is a genuine reversible reaction, and reaches an equilibrium at 80—85% of hydrolysed maltose. Maltose, revertose, and gentiobiose are the products of the action of yeast extract on dextrose. C. RANKEN.

Examination of very old cold-stored hops. A. C. CHAPMAN (J. Inst. Brew., 1926, **32**, 574).—Alameda (Californian) hops of the 1896 growth and placed in the cold store in Feb., 1897, gave on analysis in Dec., 1926, the following results: moisture 12·05%, soft resins 10·0%, hard resins 8·8%, and tannin 2·67%. The preservative power determined by the Chapman biological method (cf. B., 1925, 185) and by the lead precipitation method of Ford and Tait (cf. B., 1924, 568) gave the values 26 and 32, respectively. The α-acid had completely disappeared and the whole of the preservative property was resident in the β-resin. C. RANKEN.

Vitamin content of grapes and grape wines. A. MERJANIAN (Z. Unters. Lebensm., 1926, **52**, 307—311).—Bezssonov's vitamin test was applied to grape juice and grape products. Positive tests were given by grape juice and some wines. The vitamin content of wines appears to decrease with age. Wine yeasts contained no vitamin and the must only minute quantities. Traces of vitamin were detected in pure wine vinegar.

A. G. POLLARD.

Boiling point apparatus for determining strength of wines. G. FILAUDEAU (Ann. Falsif., 1926, **19**, 531—536).—The Malligand ebullioscope gives satisfactory indications of the alcoholic strength of wines.

D. G. HEWER.

Determination of alcohol in spirits by distillation. E. FEDER and L. RATH (Z. Unters. Lebensm., 1926, **52**, 292—294).—In the distillation process for the determination of alcohol, certain losses of alcohol occur when the liquid under examination has a high alcohol content. This was traced to the carrying over of uncondensed alcohol by expelled air during the initial warming up of the apparatus, and could only be trapped by immersing the end of the condenser in water. A simple method of arranging a pyknometer and funnel for this purpose was successfully adopted. Results obtained in this manner were approximately 0·1% higher than by the official (German) method, and were more closely in agreement with figures calculated from the density of the extract-free liquor (Berg).

A. G. POLLARD.

Preparation of dilute alcohol and liquids of definite alcoholic strength, and methods of determining alcohol. G. Tellera (Atti R. Accad. Lincei, 1926, **65**, 705—710).—Formulæ are given for calculating the proportions of water to be added to aqueous alcohol or to a liquor having a certain alcoholic content to reduce it to any required content. For the determination of alcohol, Chabot's method (B., 1926, 210; see also Widmark, A., 1922, ii, 789) is recommended and details of the procedure are given. T. H. Pope.

Acetic acid and vinegar. Kreutz and Büchner. —See XX.

Patents.

Manufacture of bacterial preparations. M. L. Landman (E.P. 261,455, 7.8.25).—A solution of 1 pt. of agar-agar in 24 pts. of water is gradually mixed with an equal volume of heavy mineral oil. A milk or whey-broth culture of the bacilli is then stirred in until an emulsion is formed, with the oil as the continuous phase, and the aqueous solution and the culture the disperse phase (for *Bacillus acidophilus* the distribution in the final product should be about 350 million/c.c.). F. R. Ennos.

Method of making plant foods (U.S.P. 1,606,015).—See XVI.

Manufacture of butter fat products (U.S.P. 1,605,009).—See XIX.

XIX.—FOODS.

Colours in foodstuffs. H. Drake-Law (J.S.C.I., 1926, **45**, 428—434 T).—The essential properties of foodstuff colours are described, and reference is made to the different causes of harmfulness. Toxicity, antisepticity, the irritating action on the skin are mentioned, and the dangerous limits of harmful metallic impurities are given. The various properties required for confectioners' colours are outlined, such as fastness to light, heat, acids, alkalis, sulphur dioxide, and metals. The methods used for testing are described. The essential properties of the best known colours are given in detail, and the uses to which they are applied are set forth, special attention being given to export trade. An extensive list of colours which are now used, or which have been used in the past, is shown, and the best known methods of identification of these are fully described. Consideration is given to the diluents commonly associated with confectioners' colours, and the method of testing for strength of diluted colours is outlined. The manner of determining the chief metallic impurities is explained, and reference is made to the many processes of analysis now in common use. The need for a standard method of analysis of all confectioners' products is emphasised, especially in connexion with export trade. Attention is directed to the growing necessity for the international standardisation of methods of analysis of the raw materials used in foodstuffs, especially in view of the more stringent foreign regulations now being imposed on imported goods.

Influence of peptic digestion in the determination of total carbohydrates in cereal products. B. G. Hartmann and F. Hillig (J. Assoc. Off. Agric. Chem., 1926, **9**, 482—484).—In the case of products containing large amounts of proteinaceous matter, the determination of carbohydrates by conversion into dextrose by means of diastase and acid hydrolysis is slow and not always reliable. By digesting the cereal with pepsin at room temperature overnight, boiling, cooling to 65°, and adding malt infusion, the conversion into dextrose is usually complete in 30 min. F. R. Ennos.

Function of oils and fats, and emulsions of oil and water in breadmaking with special reference to gluten formation and modification. E. B. Bennion (J.S.C.I., 1926, **45**, 435—438 T).—Oil and emulsions of oil and water were used in gluten washing determination, and it was found that there is a distinct action, especially on grades of flour containing a high natural percentage of gluten, such as American flour and straight-run home-milled flours. Emulsions of oil in water increase the amount of gluten in the flours to a slightly less extent than oil itself. The colour of the glutens, dried in hot-air ovens at 105° to constant weight, which were obtained from doughs made up with emulsions of oil in water was of a much lighter character, being a creamy shade instead of dark brown as normally. A standard method of gluten determination is given which was employed in the research. Oil and emulsions of oil retarded fermentation to a slight extent as judged by cylinder tests. Baking tests were carried out, and the loaves containing the emulsion possessed the best coloured crumb and pile, the colour being whiter than the crumb of the loaf containing oil alone, and much whiter than the control loaf. Oil will keep the crumb of a loaf moist, but an emulsion is more effective. A table of moisture contents of loaves made by different methods is given. The use of emulsions in the manufacture of gluten bread is also considered.

Differentiation between flours by means of iodine absorption and rate of sedimentation. N. A. Trofimuk (Z. Unters. Lebensm., 1926, **52**, 311—318).—The nature of flours is definitely indicated by the iodine adsorption of their aqueous extracts. The filtered extracts are titrated with iodine solution in the presence of starch to match a standard colour. The cereal flours can readily be distinguished from each other by this means. A marked difference exists between the range of iodine absorption of flours from mono- and di-cotyledonous seeds. Somewhat similar differences occur in the rates of sedimentation of various flours. Measurements are made of the rates of increase of depth of sediment in a cylinder of flour suspension. Admixture of 10% of rye, of 15% of oat, or of 20% of barley flour to wheaten flour may be detected in this way. A. G. Pollard.

[Phosphoric] acid in potato flour and syrup and its determination. H. Tryller (Z. Spiritusind., 1926, **49**, 342—343, 355—356, 365—366; cf. B., 1925, 221).—As contrasted with prima potato flour, no definite conclusions as to the acidity of the secondary and tertiary flours can be drawn from titration with sodium hydroxide and phenolphthalein and with hydrochloric acid and methyl orange. The acidity is greater than is accounted for by the phosphoric acid in the ash and is probably due to amino-acids derived from albuminous matter. Comparison of acidities is best made by determination

of the hydrogen-ion concentrations. The p_H value of a starch is the same as that of the liquid with which it is in equilibrium. For the prima flour the liquid used is a buffer solution consisting of 25 c.c. of a mixture of 6 pts. of $0 \cdot 1M$-disodium phosphate and 4 pts. of $0 \cdot 1N$-monopotassium phosphate diluted to 1 litre, but for the less pure secondary and tertiary flours distilled water only is required. The hydrogen-ion concentration is then determined in the liquid by the colorimetric or the electrometric method. In the case of the prima flour the p_H value may also be arrived at by titration with acid and alkali, all three methods giving concordant results. Comparison of the electrometric neutralisation curves of the prima flour with those of various acids, confirms the fact already established that the acid present is entirely phosphoric, and that it is not combined with starch as an ester. The curves for the secondary and tertiary flours show no similarity to that of phosphoric acid, but resemble either that of albumen from the sludge or of the amino-acid glycocoll. By determining the p_H values of successive wash waters it is possible to follow the course of the washing of starch free from acid, and to determine the exact point at which to stop to obtain a neutral product. The physico-chemical method is also applicable to the syrup. In order to get concordant p_H values it is necessary to work with solutions of the same concentration, approximating to 1 pt. of syrup in 2—4 pts. of water. F. R. ENNOS.

Determination of fat in malted milk. E. S. ROSE (Amer. J. Pharm., 1926, 98, 595—596).—A modified Werner–Schmid method in which a mixture of 2 pts. of "benzin U.S.P." and 1 pt. of washed ether is used as the extraction medium. F. R. ENNOS.

Preservation of milk samples with formaldehyde and trioxymethylene. LETOURNEUR-HUGON and VALIN (Ann. Falsif., 1926, 19, 544—547).—Two drops of 40% formaldehyde and about 1 g. of trioxymethylene are used to preserve about 250 c.c. of milk with more satisfactory results than with dichromate, and with entire elimination of gaseous fermentation. Such preserved samples may be analysed by ordinary methods, except that lactose should be determined polarimetrically and not by Fehling's solution. In using the Gerber method for fat it is necessary to heat at 70—80° on a water bath for some time before centrifuging to ensure re-solution of the coagulum. The Adam method is not suitable. D. G. HEWER.

Physico-chemical basis of the density of milk. G. KOESTLER (Z. Unters. Lebensm., 1926, 52, 279—287).—The density of milk can be represented as a mathematical summation, based on the densities of its constituents determined in the dispersed colloidal condition. Methods for determining these values are outlined. Mean values recorded are: d_{15}^{15} for fat, $0 \cdot 920$; dicalcium caseinate, $1 \cdot 450$; lactose, $1 \cdot 607$; salts, $2 \cdot 619$. Using these values, calculated and experimental values for the densities of a number of milk samples were in much closer agreement than when the usually accepted values of Fleischmann were employed. For a sample of "pathological" milk, calculated and observed values were more divergent. A. G. POLLARD.

Determination of the freshness of eggs. E. DINSLAGE and O. WINDHAUSEN (Z. Unters. Lebensm., 1926, 52, 288—291).—Alterations in the weight and density of eggs with time depend upon the method of storage, temperature, and moisture content of the surrounding atmosphere. The rate of decrease of density of eggs with age is lowered by increased moisture content of the surrounding air. In the absence of information as to method of storing and the original density of the egg, it is impossible to decide its age with any reasonable degree of accuracy. A. G. POLLARD.

Specific characters of regenerated preserved peas. J. FROIDEVAUX (Ann. Falsif., 1926, 19, 536—544).—The proportion of the water-insoluble portion of dried peas was found to be unaffected by the temperature of sterilisation employed, and by the method of preparation previous to boxing. The commercial value of preserved peas is inversely proportional to the amount of desiccation before boxing, and quality is higher the lower the nitrogen and higher the water content in the insoluble portion. D. G. HEWER.

Composition and nutritive value of pumpkin-seed cake. A. ZAITSCHEK and E. JALOWETZ (Kisérlet. Közlem., 1924, 27, 26—31).—Low-grade cake contained 41% of crude fibre (8% digestible); high-grade, 21% (none digestible). A large number of samples contained 40—50% of protein, 11—30% of fat, and had a starch value of approximately 90%. A. A. ELDRIDGE.

Vitamin-containing food preparations and their content of anti-beri-beri vitamin. E. KOMM (Z. Unters. Lebensm., 1926, 52, 303—307).—Animal nutrition experiments are described indicating the presence of vitamin-*B* in a number of proprietary food preparations. A. G. POLLARD.

Viscosity of pectin sols. A. OHN (Ind. Eng. Chem., 1926, 18, 1295—1298).—A pectin preparation obtained by alcoholic precipitation from a water extract of the white inner rind of oranges turns yellow at 155°, decomp. 158°, has d^{20} $1 \cdot 180$, and is precipitated by soluble copper and lead salts. The relative viscosity of sols made from this preparation, citric acid, cane sugar, and distilled water depends upon the proportions of these constituents, but, within limits and when the hydrogen-ion concentration is sufficient, an increase in the amount of pectin or sugar gives a noticeable increase in relative viscosity. If the pectin is deficient, increase in the other factors gives no appreciable change in the relative viscosity. The optimum ratio for a good jelly is $0 \cdot 40$ g. of pectin and $62 \cdot 5$ g. of sugar per 100 c.c. at p_H $2 \cdot 60$, the relative viscosity of this sol at 103° then being $0 \cdot 560$, compared with a glycerol standard (d^{20} $1 \cdot 220$) at 60°. There is a large increase in relative viscosity below 90° of sols having sufficient pectin, acid, and sugar, this increase being greater below 70°. Within limits the surface tension of these sols varies inversely as the viscosity, this variation depending mainly on the hydrogen-ion concentration. E. HOLMES.

Apparatus for the determination of moisture [in butter] by distillation with xylene. PRITZKER and JUNGKUNZ.—See I.

Lacquers for preserve cans. SERGER.—See XIII.

Acetic acid and vinegar. KREUTZ and BÜCHNER.—See XX.

Citrus flavours. SCHUETTE and DOMOGALLA.—See XII.

PATENTS.

Process of producing edible fat. G. GRINDROD, Assr. to CARNATION MILK PRODUCTS Co. (U.S.P. 1,605,108, 2.11.26. Appl., 12.5.19).—Edible vegetable fats are hydrogenated, in carefully-regulated stages, to remove toxic constituents and substances likely to produce rancidity, and to avoid hardening of the fat. The material is first hydrogenated in the presence of a catalyst at 70° to decomposed aldehydes and ketones, and then under 250—600 lb. pressure at 90° to decompose the hydroxy-fats. F. R. ENNOS.

Manufacture of butter fat products. T. A. SPAETH, Assr. to MATTHEWS SELECTED DAIRIES Co. (U.S.P. 1,605,009, 2.11.26. Appl., 1.5.26).—Sweet cream is mixed with a lactic culture at about 24°, and an acidity of approximately 0·9% of lactic acid allowed to develop. A smaller quantity of a solution of gelatin in milk is mixed with the acidified cream and the whole is left in the cool for about 12 hrs. No further souring will occur for an extended period. F. R. ENNOS.

Preservation of eggs. A. J. BELLAMY, and A. BELLAMY & Co., LTD. (E.P. 261,194, 13.5.25).—After removal of the shells, the remainder or the yolk only is mixed with 2½—15% of glycerin, and the mixture concentrated in shallow trays by exposure to dry air until the pasty residue contains not more than 40% by weight of moisture. F. R. ENNOS.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Identification of acetic acid in lavender oil. P. LANGLAIS and J. GOBY (Perf. Essent. Oil Rec., 1926, **17**, 520—521).—Examination of a blended lavender oil (*d* 0·892, α_D −6° 20′, ester number 121·42, esters calculated as linalyl acetate 42·5%) has shown that, contrary to the statements of Dalton (*ibid.*, 432—3, 476—7), the predominating acid is acetic acid. By incomplete steam-distillation of the acidified potassium salts obtained on saponification, acetic acid corresponding to 9·2% of the oil was isolated and identified by conversion into acetanilide, *p*-bromoacetanilide, and ethyl acetate. E. H. SHARPLES.

Acetic acid and vinegars containing formic acid. A. KREUTZ and C. BÜCHNER (Z. Unters. Lebensm., 1926, **52**, 295—298).—The popular prejudice against synthetic vinegars containing acetic acid prepared by the carbide process is unfounded. The amount of formic acid present in such vinegars is too small to be injurious. Determination of formic acid in vinegar by the reduction of permanganate is not indicative of the source of the vinegar. Many artificial vinegars are coloured with burnt sugar. The latter contains reducing substances which render the permanganate test for formic acid valueless. A. G. POLLARD.

Detection of alcohol adulterants. J. M. HALEY (Ind. Eng. Chem., 1926, **18**, 1312—1313).—Detailed methods for the rapid but certain detection of methyl alcohol, acetone, benzene, alkaloids, phenol, and ethyl phthalate are given, whereby the complete test for these six adulterants may be carried out in 1 hr. E. HOLMES.

Furfuraldehyde in industry. D. H. KILLEFFER (Ind. Eng. Chem., 1926, **18**, 1217—1219).—A general account of the transition of furfuraldehyde from a laboratory curiosity to an industrial raw material, of its present and possible uses, together with a detailed description of the present commercial method of preparation. E. HOLMES.

Colour reaction of saccharin. L. EKKERT (Pharm. Zentr., 1926, **67**, 821—822).—The known colour reactions of saccharin are summarised, and the following additional reaction is described. To 0·02—0·05 g. of saccharin in 1—2 c.c. of water are added 3—6 drops of 30% hydrogen peroxide solution, followed by 0·01—0·04 g. of sodium nitrite, and the mixture is well shaken. The solution first acquires a yellow colour, which is changed on addition of alkali into a vivid brown-red. The reaction is also given by other phenols, such as resorcinol, and by morphine, but these substances give a yellow solution on treatment with sodium nitrite and dilute sulphuric acid, whilst saccharin under the same conditions yields a colourless solution. W. J. POWELL.

[Assay of] *Cascara sagrada*. J. B. BERARDI and M. C. CANAN (J. Amer. Pharm. Assoc., 1926, **15**, 189—193).—On extraction of fluid extracts of cascara, aloes, frangula, or rhubarb with ethyl ether or ethyl acetate, the residues were inert; the colours produced by the extracts with ammonia were not characteristic. Of two colour-reacting constituents in *Cascara sagrada*, one, insoluble in water, gives a deep cherry-red colour with ammonia; the other, soluble in water, gives an orange-brown colour. The non-bitter principle is soluble in water and is therapeutically active. The bitter principle is soluble in water, accompanies or is the brown-colour-reacting constituent and accompanies or is part of the therapeutically active principle, probably an anthraquinone derivative. A. A. ELDRIDGE.

Industrial conversion of coal into organic products of technical use. PATART.—See II.

PATENTS.

Manufacture of solvents [from petroleum hydrocarbons]. C. P. BYRNES (E.P. 259,293, 6.7.25).—The partial oxidation products of petroleum hydrocarbons, obtained as in E.P. 138,113 (B., 1921, 636), 173,750 (B., 1923, 818), and 209,128 (B., 1924, 210), are extracted with mixtures of alcohol and water in such proportions that the more highly oxidised components (esters, aldehydes, and ketones) are removed. The alcohol is distilled off, preferably after dehydrating the solution with, *e.g.*, anhydrous sodium sulphate, and the residual oil is fractionated. The fractions boiling from 100° to about 250° are suitable solvents for nitrocellulose and varnish gums in the preparation of lacquers and lacquer paints. Any free acids present in the partially oxidised oils may be esterified, before the extraction with aqueous alcohol, by addition of a suitable catalyst, such as hydrogen chloride. Again, all or part of the aldehydes or ketones may be removed from the alcoholic solution by extraction with sodium bisulphite. A. DAVIDSON.

Vanadium compound [of 8-hydroxyquinoline]. H. HAHL and W. KROPP, Assrs. to WINTHROP CHEM. Co., INC. (U.S.P. 1,607,196, 16.11.26. Appl., 25.9.25. Conv.

24.12.24).—8-Hydroxyquinoline (30 pts.) and vanadium trioxide (10 pts.) are heated with water (300 pts.), and the brownish-green powder obtained is washed, and dried. It has the probable formula $V(OC_9H_6N)_3$ and is of therapeutic value in the treatment of spirochætal diseases. Other oxides of vanadium and derivatives of 8-hydroxyquinoline can also be employed.
T. S. Wheeler.

Manufacture of acyl halides. Brit. Dyestuffs Corp., Ltd., and S. Coffey (E.P. 261,240, 15.3.26).—Acyl halides are prepared pure and in good yield by treating a mixture of fatty acid and anhydride with a phosphorus trihalide. No hydrogen halide is formed. *E.g.*, a mixture of 27·3 pts. of 100% acetic acid, 18·7 pts. of acetic anhydride, and 35·5 pts. of phosphorus trichloride is warmed. An 85% yield of pure acetyl chloride distils over.
B. Fullman.

Disinfectant, antiseptic, and medicament. Rhenania Verein Chem. Fabr. A.-G., Assees. of O. Gerngross and K. Rülke (U.S.P. 1,606,359, 9.11.26. Appl., 2.1.25. Conv., 28.1.24).—The disinfectant properties of heavy metal salts, such as mercuric chloride, mercuric thiocyanate, or silver cyanide, are greatly increased by the addition of a large excess of an alkali or alkaline-earth thiocyanate. Suitable mixtures, of special value in disinfecting albuminoid matter, comprise mercuric thiocyanate (1 pt.) and potassium thiocyanate (124 pts.), or potassium silver cyanide (1 pt.) and potassium thiocyanate (99 pts.), each mixture being used in about 3% solutions.
T. S. Wheeler.

Making *m*-nitro-*p*-hydroxyphenylarsinic acid. I. Ostromislensky, Assr. to Ostro Products Corp. (U.S.P. 1,607,299, 16.11.26. Appl., 22.7.22).—A mixture of arsanilic acid (*p*-aminophenylarsinic acid; 250 g.), nitric acid *d* 1·3 (275 c.c.), and water (750 c.c.) is treated with sodium nitrite (133 g.), the temperature being maintained at 70°. The mixture is then heated near its b.p. under reflux for 6—9 hrs. Diazotisation, replacement of the diazo-group by hydroxyl, and nitration successively occur, and on cooling *m*-nitro-*p*-hydroxyphenylarsinic acid separates in 80% yield. It can be purified by dissolving in boiling 15% sodium hydroxide solution, and precipitating the sodium salt with ethyl alcohol.
T. S. Wheeler.

Treating tobacco. J. W. Kelland (E.P. 261,520, 16.4.26).—Tobacco waste (stalk, offal, etc.) is softened, pulped, and kneaded until the fibres are reduced to about 1 mm. in length. The pulp is then made into sheets or slabs, 33% acetic acid solution being added for preservative purposes if necessary.
B. Fullman.

Manufacture of bacterial preparations (E.P. 261,455).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Patents.

Reducing photographic negatives, positives, films, bromide, and gaslight prints. K. E. Taeschner (E.P. 261,201, 7.12.25).—Reduction is effected by bathing the image in a solution of iodine and potassium iodide, and either simultaneously or previously, with a solution of alkali chromate or dichromate. The reducing solution may also contain sugar. Tanning of the gelatin in the vicinity of the silver by the chromate or dichromate protects the lighter tones from the action of the iodine.
W. Clark.

Photographic papers for making prints from tracings. H. Kammerer (E.P. 252,012, 7.5.26. Conv., 7.5.25).—Light-sensitive papers producing positive pictures directly by the action of aromatic amines, phenols, or aminophenols, are produced by coating with a chromate or dichromate solution containing heavy metal salts of organic acids, especially ferric salts such as ferric ammonium citrate, oxalate, or tartrate, to which may be added a salt of manganese, or mixtures of such salts. The amines etc. required for development may be applied to the paper before exposure in a dry state or in solution in spirit, development being by means of a damp sponge, or by steam. Paper prepared in the manner described keeps much better than that usually prepared for the aniline process.
W. Clark.

Manufacture of diffraction gratings for spectroscopic, optical, and like purposes. K. Kondo (E.P. 262,020, 29.7.26).—A replica of a diffraction grating is obtained by pressing the original against a plane dry plate coated with bichromated gelatin, suitably exposing to light, developing the plate in hot water, and silvering. An original plane grating is reproduced on a concave plate in the above manner by employing a parallel beam of light. Gratings suitable for investigating the ultra-violet region are made by electrolytically depositing nickel on the silver coating of concave gratings prepared as above.
A. Cousen.

***X*-Ray screens.** H. S. Sawford (E.P. 255,831, 15.6.26. Conv., 24.7.25).

XXII.—EXPLOSIVES; MATCHES.

Nitrometric determination of tetryl. L. Desvergnes (Ann. Chim. Analyt., 1926, [ii], **8**, 353—357).—Tests of Lehmstedt's method for the determination of tetryl by measuring the nitric oxide evolved on reduction with mercury and sulphuric acid (B., 1926, 388) always gave very low results when pure tetryl was used. Tetryl is insoluble in cold 90—100% sulphuric acid, and the reduction of 0·3 g. requires at least 4 hrs., whilst at 120° partial decomposition takes place with the formation of trinitrophenol, ammonia, and carbon dioxide. 2:4:6-Trinitrophenylethylnitroamine behaves in an analogous manner, so that the method is untrustworthy for the determination of either compound.
A. R. Powell.

XXIII.—SANITATION; WATER PURIFICATION.

Purification of sewage by activated sludge at Essen-Rellinghausen. F. Sierp (Z. angew. Chem., 1926, **39**, 1521—1534).—A detailed account is given of experiments started in 1912 which have led to the installation of an activated sludge plant for a sewage containing acid waste. It was found that mechanical agitation was more effective and less costly than aeration alone, as the former brings the sludge more into contact with the sewage and the colloid-absorbing properties of the sludge can be utilised to the greatest advantage. Aerobic conditions, however, are essential and now aeration is combined with agitation, the air being introduced through filter plates as a fine blast, this having been found more effective than through perforated lead pipes. The speed

of agitation is important; it must be fast enough for the sludge to be broken up, but not so fast that the sludge does not settle to the bottom and so escapes the action of the air. Paddles rotating 7 times per minute are found to be most effective. For good working of the process the condition of the sludge must be watched so that its flocculating properties are utilised, and the extent of oxidation of the effluent must be tested. Great importance is attached to the breaking of the surface film by agitation to ensure direct absorption of the air. The working of the plant is also watched by determining the "oxygen consumed," by noting the transparency of the effluent, and by the free-ammonia decrease, and ratio of nitrite to nitrate. Recently, hoods have been provided for collecting the gas evolved, which is subsequently burnt. W. G. CAREY.

Biochemical oxygen demand of raw and treated sewage. C. E. KEEFER and R. T. REGESTER (Eng. News-Rec., 1926, **97**, 870—873).—A study of several thousand Baltimore sewage analyses to ascertain the relationship existing between the biochemical oxygen demand and the solids in raw and treated sewage collected in a separate system of sewers. Generally, the oxygen demand of the sewage is low during the winter and gradually increases until the summer months. The oxygen demand of sewage and sedimentation tank effluent increases with the suspended and settling solids, but, in the case of the former, the rate of increase is less as the amount of solids becomes greater. One explanation for this condition is that as the solids in the sewage increase, the relative amount of inorganic to organic material is greater. Settling tanks lower the oxygen demand about as much as the trickling filters; during 1919—25, the settling tanks reduced the oxygen demand from 174 to 104 p.p.m., and the trickling filters from 104 to 29 p.p.m., or 70 and 75 p.p.m., respectively. 40·3% of the oxygen demand is removed by settling tanks, 43% by trickling filters, and 0·2% by humus tanks. Such figures, however, do not give a complete and comparative picture of the rôle played by the three treatment units in purifying sewage, the trickling filters being considerably more efficacious than the settling tanks, in that large volumes of dissolved and nitrate oxygen are added to the sewage. A study, having regard to the change in oxygen content as well as to the reduction in oxygen demand, of the comparative efficiencies of the settling tanks, trickling filters, and humus tanks of eleven sewage works, showed the comparative efficiencies to be 33·5%, 62·3%, and 4·2%, respectively.
W. T. LOCKETT.

Biochemical oxygen demand test. R. E. GREENFIELD, A. L. ELDER, and R. E. MCMURRAY (Ind. Eng. Chem., 1926, **18**, 1276—1279).—During the absorption of oxygen at 2° by sewage diluted with distilled water the bacterial count first increases slowly, the absorption also being very slow (B., 1926, 422), then both increase much more rapidly. If the sewage is initially heavily inoculated with the culture of the organisms the period of slow absorption is absent, so that it seems that, as previously suggested (*loc. cit.*), the lag period in the absorption is due to the slowness of the development, at the prevailing low temperature, of the bacteria effecting deoxygenation. Polluted stream water yields absorption curves showing a second period of acceleration, which does not appear, however, to be due to the death of plankton organisms alive at the commencement of the absorption, since it is not eliminated by destroying the plankton by heat at the outset. Experiments with sewage diluted at p_H 7·7 with water containing inorganic salts gave two-stage absorption curves, and a much increased oxygen demand. In the second period nitritation, which hardly occurs at all in absence of inorganic salts, was rapid and extensive. Reduction of the p_H of the diluted sewage to 6·4 decreased the oxygen demand, but a rise to 8·1 had little effect.
R. CUTHILL.

Effect of hydrogen-ion concentration in revivifying zeolites. O. R. SWEENEY and R. RILEY (Ind. Eng. Chem., 1926, **18**, 1214—1216).—The base-exchange capacity of zeolites used for water-softening is nearly always influenced—usually adversely—to a considerable extent by the presence of traces of acid or alkali in the sodium chloride solution used for revivification. This phenomenon does not appear, however, to be due to a solvent action. The p_H of the water being softened also seems to be of considerable importance. R. CUTHILL.

PATENTS.

Compositions for exterminating insects and weeds. GRAESSER-MONSANTO CHEMICAL WORKS, LTD., and H. MAXWELL-LEFROY (E.P. 261,241, 14.5.25).—Insects, flies, fly larvæ, and weeds in ships, houses, gardens, etc., may be exterminated by direct exposure to a composition prepared by emulsifying monochloronaphthalene (or monobromonaphthalene) with water with the aid of an agent, *e.g.*, soft soap. For the extermination of insects in fabrics a 10% solution of monochloronaphthalene in benzene may be used, for insects on ponds or solid surfaces, a 10% solution of monochloronaphthalene in a paraffin hydrocarbon of high flash point, and for insects on sheep, a mixture composed of monochloronaphthalene (10 pts.) and ordinary sheep dip (90 pts.), or a spray consisting of a mixture of monochloronaphthalene and a soothing oil, *e.g.*, cottonseed oil.
W. T. LOCKETT.

Extermination of insects. GRAESSER-MONSANTO CHEMICAL WORKS, LTD., and H. MAXWELL-LEFROY (E.P. 261,055, 14.5.25).—A lethal concentration of monochloronaphthalene or monobromonaphthalene is produced locally or within the space to be treated, by volatilisation of the compound by heat from a solid body, *e.g.*, kieselguhr impregnated with the liquid, or by atomising a mixture of the monohalogenated naphthalene and a hydrocarbon oil of high flash point. The monohalogenated naphthalenes are specific poisons for insects, and especially flies and fly larvæ, even at such low concentrations as one part by volume per million parts of air.
W. T. LOCKETT.

Method of fumigation [with hydrogen cyanide]. W. S. LANDIS and G. H. BUCHANAN, Assrs. to AMERICAN CYANAMID CO. (U.S.P. 1,607,261, 16.11.26. Appl., 8.7.21).—The removal of hydrogen cyanide from a space which it has been employed to fumigate, is accomplished by injecting into it a mixture of formaldehyde and steam.
T. S. WHEELER.

Method and apparatus for treating refuse. F. C. EVANS (E.P. 261,119, 24.8.25).

Disinfectant, antiseptic, and medicament (U.S.P. 1,606,359).—See XX.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

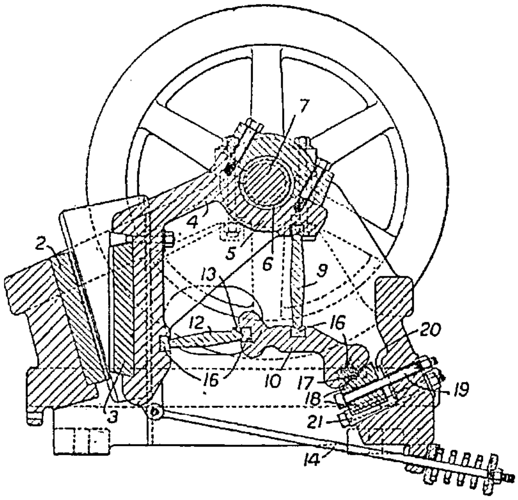
FEBRUARY 18, 1927.

I.—GENERAL; PLANT; MACHINERY.

Catalytic power of [animal] charcoal. V. Podrouzek (Chem. Listy, 1926, **20**, 553—555).—The catalytic activity of animal charcoal depends on its iron content; extraction of the charcoal with hydrochloric acid reduces its activity practically to zero when all the iron has been removed. Absolutely pure carbon, whether in coarse particles or in colloidal solution, shows no sign of catalytic activity. A. R. Powell.

Patents.

[Jaw] crushing machines. E. O. Stubbings (E.P. 261,211, 1.1.26).—As shown in the figure, a single eccentric, 7, reciprocates the upper part, 4, of the jaw



directly, and gives an exaggerated motion to the lower part, 3, of the jaw by means of the toggles, 9, 10, and 12. A sliding wedge piece, 18, is provided to take up wear. B. M. Venables.

[Fine] grinding mill. J. Ehrhardt (E.P. 261,322, 8.9.26).—An apparatus for fine grinding comprises a disc rotating preferably on a vertical shaft, against the edge of which a number of blocks are individually pressed by a weighted lever and screw. Entrance for the feed material is provided by passages formed in the blocks, emerging near the leading edge, and the ground material falls away between the blocks. By setting the axes of the blocks at an angle to their respective radii a larger proportion of the edge of the disc is rendered effective. B. M. Venables.

Regenerative heat-exchanging apparatus. E. H., and E. P. Kignell (E.P. 249,152, 12.3.26. Conv., 14.3.25).—A heat exchanger for two or more fluids is arranged so that the conduit for one fluid forms the container for the heat-transferring substance, which has passages left in it for the flow of the fluids. The other fluid or fluids enter and leave through the side of the main conduit, and all the fluids are distributed to the various passages of the regenerator in turn by continuously rotating valves. B. M. Venables.

Refrigerating processes. R. W. Davenport (E.P. 249,097, 25.2.26. Conv., 12.3.25).—The refrigerant is circulated continuously in a closed cycle, and comprises a condensible component, such as carbon tetrachloride, and a non-condensible component insoluble in and inert to the condensate, such as air. The latter promotes normal boiling, *i.e.*, without superheating. The cycle includes compression of the gas and vapour together, cooling of the compressed mixture to condense part of the vapour, expansion of the gas and vapour in thermal contact with the liquid, and heating of the liquid to vaporise it in contact with the gas. The non-condensible component is of sufficient volume to produce a total pressure on the liquid substantially exceeding its own vapour pressure, and such volume is determined in accordance with the temperature desired in the refrigerating zone. The total pressures of the high and low sides of the cycle remain constant, and the ratio of the partial pressure of the non-condensible component to the total pressure is kept constant. H. Holmes.

Evaporating apparatus. D. A. Blair, and Blair, Campbell & McLean Ltd. (E.P. 262,608, 30.12.25).—A system of evaporation (especially suitable for tannin extracts) comprises a film evaporator combined with at least two other evaporators which contain a good bulk of liquid. The film evaporator does the bulk of the evaporation continuously, and the somewhat irregular product from it is collected in one of the bulk evaporators while the other bulk evaporator is finishing a batch. B. M. Venables.

Process of evaporation. W. L. Badger, Assr. to Swenson Evaporator Co. (U.S.P. 1,609,853, 7.12.26. Appl., 9.8.23).—A crystallisable liquid is boiled in an evaporating chamber, and small quantities are withdrawn and superheated. The superheated liquid is re-introduced into the chamber, where it flashes, producing seed crystals which promote the crystallisation. H. Holmes.

Apparatus for drying granular materials. F. M. Parker (E.P. 261,517, 3.10.25).—The material is subjected to a countercurrent of furnace or other hot gases as it slides down a zigzag passage constituted by superposed compartments carried by a connecting frame, the movement of the material being produced by imparting a swinging motion to the frame. The compartments may be inclined tubes communicating at their ends, and partitioned longitudinally to provide different passages for different grades of material. Alternatively, a casing carried by the frame may be divided into compartments by trays attached alternately to the opposite end walls,

each tray sloping downwards from the point of attachment and terminating at a distance from the other end wall. The dryer may be enclosed to conserve heat radiated from the top of the furnace, and the receiving end of the discharge shoot for the material may be mounted within the furnace. H. HOLMES.

Mixing process and apparatus. J. W. SMITH (E.P. 261.135, 4.9.25).—Flowable materials are mixed by passing them through a conduit which is provided with a number of deflecting devices, each comprising a ring of tapered fingers or blades extending from the circumference inwardly and inclined. The blades are preferably twisted to a helicoidal shape so as to give a whirling motion to the liquid, which is reversed at alternate mixing devices. B. M. VENABLES.

Centrifugal mixing or emulsifying apparatus. F. GRIMBLE, M. N. CAIRD, and E. COOMBS (E.P. 261,904, 7.11.25).—The apparatus comprises a number of specially-shaped discs assembled on a hollow shaft or driver which forms the feed passage. The outlet spaces between the discs are segmental (*i.e.*, not complete annuli), and are narrower at the outer circumference. Corrugated material may also be placed between the discs with the corrugations substantially radial.

B. M. VENABLES.

Separation of the constituents of gaseous mixtures. Liquefaction and separation of gaseous mixtures. C. C. VAN NUYS, Assr. to AIR REDUCTION Co., INC. (U.S.P. 1,607,320—1, 16.11.26. Appl. [A] 2.8.21; [B] 31.1.23).—(A) A constituent not readily liquefiable is separated by subjecting the mixture to selective backward-return condensation, first with previously liquefied portions evaporating at a lower pressure, and afterwards with an extraneous liquid evaporating at a lower temperature, thus liquefying selectively substantially all but the desired constituent. (B) A portion of the mixture is partially expanded. Another portion is liquefied by indirect contact with a liquid enriched in the less volatile constituent, and the liquefied mixture is rectified by direct contact with the partially-expanded gaseous portion. The gases resulting from the rectification are subjected to selective liquefaction with backward return to supplement the liquid enriched in the less volatile constituent produced by the rectification. H. HOLMES.

Liquefaction of gases. C. C. VAN NUYS and J. L. SCHLITT, Assrs. to AIR REDUCTION Co., INC. (U.S.P. 1,607,322, 16.11.26. Appl., 10.1.25).—The gas is compressed, cooled, partially expanded, and further cooled to the liquefaction temperature corresponding to the expansion pressure. It is then subjected to backward return condensation by thermal contact with previously liquefied portions. H. HOLMES.

Separation of the constituents of gaseous mixtures. C. C. VAN NUYS, Assr. to AIR REDUCTION Co., INC. (U.S.P. 1,607,708, 23.11.26. Appl., 22.10.24).—The mixture is subjected to a primary rectification to produce a gas impoverished in one constituent. This gas is withdrawn, recompressed, and liquefied, and the liquid is subjected to an auxiliary rectification to separate another constituent in substantial purity. The low temperature of the auxiliary rectification is maintained by application of refrigeration from the primary rectification. H. HOLMES.

Liquefaction and rectification of gases. C. C. VAN NUYS, Assr. to AIR REDUCTION Co., INC. (U.S.P. 1,609,450, 7.12.26. Appl., 2.12.19).—A gaseous mixture is liquefied to separate a portion of one constituent as a residual gas which is withdrawn, expanded, and caused to travel in indirect contact with the mixture to be liquefied. The liquid is rectified to produce a liquid comprising one constituent in substantial purity, and an effluent having substantially the composition of the original gaseous mixture. The liquid is vaporised, and the vapour is withdrawn separately from the effluent.

H. HOLMES.

Method and means for determining or comparing the viscosities of fluids. KNOW MILL PRINTING Co., LTD., and T. L. MORT (E.P. 262,539, 18.9.25).—The apparatus comprises a handle provided at the end with a depression or cup accurately ground to fit a ball. The distance between the ball and the cup is regulated by means of a screw within the handle terminating in three small feet at the centre of the cup. To operate, the ball is pressed into the cup when both are below the surface of the liquid to be tested, and the time taken for the ball to fall away under its own weight is noted when the gap is set at a standard thickness; or the gap may be adjusted till the ball falls away in standard time.

B. M. VENABLES.

Viscosity meters. C. M. LARSON and C. L. KNOPF (E.P. 262,652, 17.5.26).—Two similar tubes, each containing a ball or other solid particle, the one filled with a standard liquid (which may be sealed) and the other filled with the liquid to be tested, are tilted together and the time noted for the ball to run down. A form of the apparatus suitable for direct attachment to the crank-case of an engine is described. B. M. VENABLES.

Filters for air and gases. D. HALL, J. H. KAY, and HALL & KAY LTD. (E.P. 261,897, 26.10.25).—The filter medium, which may be flannel or other fabric, is backed by wire netting or similar material stretched over the open frames. B. M. VENABLES.

Preparation of diatomaceous earth. R. C. WILLIAMS, Assr. to THE DIATOM INSULATION Co. (U.S.P. 1,606,281, 9.11.26. Appl., 15.7.25).—Raw diatomaceous earth is mixed with a large quantity of water and deflocculated by the addition of a suitable substance, the mixture is then flocculated by the addition of another substance and allowed to settle, the broken skeletal frames and impurities being decanted off with the water from the heavier whole skeletal frames.

B. M. VENABLES.

Lubricant. E. M. HONAN and J. R. TOWNSEND, Assrs. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,606,788, 16.11.26. Appl., 17.12.24).—Spermaceti wax and petrolatum are dissolved in a readily volatile vehicle.

H. ROYAL-DAWSON.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Coking process and path of travel of gases in the coke oven. T. SCHMIDT (Fuel, 1926, **5**, 486—509).—In a partially carbonised coke-oven charge there exists

a zone of uncarbonised coal separated from a zone of semi-coke by a layer of coal in the plastic state. As heat penetrates the charge the plastic layer is transformed into semi-coke and a new plastic layer is formed in the succeeding layer of coal. Immediately after charging an oven a considerable positive pressure, increasing towards the sole, exists in all planes of the charge. As the plastic layer advances from the oven wall the pressure within the semi-coke zone formed falls to a slight vacuum, which is fairly uniform throughout the height of the oven. The positive pressure exists, however, on the cooler side of the plastic layer so long as any uncoked coal remains. The temperature in the semi-coke rises from 500° to 1000° according to the duration of its heating. The uncoked coal remains at 100° so long as it contains any moisture, which in the case of the coal at the centre of a 21-in. oven is not driven off until 18 hours after charging. The plastic layer, therefore, is divisional between a zone of high pressure and low temperature, and one of low pressure and high temperature. Depending upon the nature of coal in the plastic state, the carbonisation in coke ovens is more or less a two-stage process. Samples of a coal which was being carbonised in ovens on the large scale were distilled up to 500° in the laboratory, and the nature and the distribution of the products were determined. A sample of freshly-formed semi-coke was taken from one of the ovens and distilled to 1000°, its products being determined and examined. The formation of tar ceases with the plastic state, and no tar was formed by distillation of the semi-coke. The permanent gas is equally distributed, by weight, between the high- and the low-temperature stages, but the relative densities and, therefore, volumes in the two cases are very different. Samples of gas from the uncoked coal 1 hr. and 12 hrs., respectively, after charging were both precisely similar to the gas obtained from the low-temperature distillation of the coal in the laboratory, indicating that little or no gas passes from the semi-coke through the plastic layer, which is to be expected from the rise in pressure across the plastic layer in that direction. Some of the low-temperature products pass from the plastic layer into the coke, as indicated by the presence of tar in samples of gas from the coke zone, and also by the high methane and low hydrogen content of the gas passing through the coke as compared with the gas distilled from freshly-formed semi-coke. It is calculated that with a lean gas coal 20—26% by volume of the permanent gas, 80% of the tar, and 10% of the ammonia produced pass through the uncoked coal. Ammonia is confined essentially to the coke zone, and is not subject to the influence of the large proportion of moisture in the coal as carbonised in coke ovens. S. PEXTON.

Relative ignition temperatures of solid fuels. K. NAKAMURA and A. SHIMOMURA (Mem. Coll. Sci. Kyoto, 1926, **A 10**, 89—94).—The relative ignition temperatures of 22 kinds of solid fuel, including various types of Japanese coal, a semi-coke, a metallurgical coke, and wood charcoal, have been determined by Wheeler's method (B., 1924, 896). The method was slightly modified by the introduction of a paraffin bath surrounding the tube containing the coal, and secondarily heated by the electrically-heated sand bath. In general, the greater the oxygen content or the volatile-matter content, or the lower the ash content of a coal, the lower is its relative ignition temperature. The rule relating to the oxygen content, however, does not apply to lignites. A. B. MANNING.

Specific gravity of lignites and of the cokes therefrom. M. DOLCH (Z. angew. Chem., 1926, **39**, 1518—1521).—The sp. gr. of a large number of brown coals and lignites rich in volatile matter from Central Germany is about 1·00, but for more ligneous brown coal it is generally lower. The desiccation of the coal with air lowers the sp. gr. of the lignites very considerably, but not that of the more pronounced ligneous coals, whilst more amorphous coals give irregular results. German brown coals shrink much less when dried in nitrogen than by air drying. The sp. gr. of the coke from various types of coal is also subject to the same variations, and here also the presence of oxygen in the drying gives a smaller coke volume with brown coal, but this is not found with coke from the more ligneous coals. The sp. gr. of coke from brown coal is about 0·50, cokes from ligneous coals are as low as 0·15, whilst cokes from more amorphous coals occupy an intermediate position. W. G. CAREY.

Wood as gas-making material. O. F. STAFFORD (Ind. Eng. Chem., 1926, **18**, 1318—1320).—Distillation of dry Douglas fir mill-waste in the generator of a water-gas plant used as an internally heated retort gave, per ton of wood, 18,000 cub. ft. of a gas of heating power 480 B.Th.U. and no tar. Substitution of wood for coal in a blue water-gas plant gave a gas very similar to that obtained when coal is used, and a little tar. Under certain conditions it appears possible to make a producer-gas of low heating power more cheaply from wood than from coal. Low-temperature distillation of wood yields relatively small amounts of a gas which is chiefly carbon dioxide, but this could be converted into a product resembling water-gas by passing it through a bed of incandescent charcoal. Soft woods are most suitable for gas-making. R. CUTHILL.

Influence of the variable composition of coke-oven gas on its economical combustion. SAUERMANN (Gas- u. Wasserfach, 1926, **69**, 1135—1141).—The composition of coke-oven gas depends on the pressure maintained in the oven, the external wind pressure and direction, the coal used, the coking temperature, and other factors, and is therefore subject to considerable variation. Analyses, calorific values, densities, volumes of products of combustion, volumes of air required for combustion, theoretical flame temperatures, etc. are tabulated for 20 samples of gas taken from a coke-oven plant at various times during the course of six months' working. No simple relation exists between the calorific values and densities of the gases. The volume of air required for combustion and the total volume of combustion products are approximately linear functions of the gross calorific value. The carbon dioxide content of the flue gases bears no relation to the calorific value, and gives no accurate measure of the excess air. The influence of the amount of excess air on the flame temperature and the heat losses, and the variation of these when a burner is supplied with gas of varying

pressure and composition, are discussed. For economical combustion good pressure regulation is necessary. At constant pressure, fluctuations in the gas supply may occur owing to variations in the density, and even at constant density the excess air in the flue gases may vary owing to the varying air requirements of the coke oven gas. For the gases tabulated, the maximum variation due to these causes is not greater than that due to a pressure difference of 25 mm. of water. Similar calculations have been made for two series of gases of varying composition, but of constant calorific value and constant density, respectively. Comparison of these shows the latter to have the advantage, gases of the same calorific value being capable of greater variation in their behaviour on combustion than gases of the same density. In evaluating a gas the density and carbon dioxide content, as well as the calorific value, must be taken into consideration. A. B. Manning.

Cracking of ozokerite. E. Toporescu (Bul. Soc. Română Stiinte, 1926, **29**, 3—5).—The cracking of ozokerite in presence of 5% of anhydrous aluminium chloride as catalyst yields products distilling at about 160°, and containing about 6% of benzine and 19% of petroleum oil. With 15% of catalyst the distillation commences at 122—125°, the distillate containing 30% of benzine and 5% of petroleum oil, and with 25% of catalyst the distillate, which first comes over at 110°, contains 7% of benzine and 2% of petroleum oil. In the last two cases large quantities of gas were evolved, and in all cases unsaturated hydrocarbons were produced. S. K. Tweedy.

Artificial ageing of mineral oils. F. Evers and R. Schmidt (Wiss. Veröff. Siemens-Konz., 1926, **5**, 210—228).—When a transformer oil is heated at 150° in an atmosphere of oxygen changes take place similar to those occurring over the course of years of use. These changes may be accelerated by dispersing the oil on silica gel previously impregnated with an oxide catalyst. The rate of oxidation of the oil is dependent on the chemical and physical nature of the catalyst, the most efficient being colloidal oxides of multivalent metals such as vanadium, molybdenum, or tungsten in combination with ferric oxide. The relative stabilities of various oils may be compared by determining the rate of oxidation under standard conditions, *e.g.*, heating for 2 hrs. at 150°, followed by a determination of the acid value of the oxidised oil. The rate of oxidation is usually very great during the first 5 min. heating, but subsequently it decreases rapidly, finally becoming stationary in 1—2 hrs. During the oxidation small amounts of carbon dioxide, carbon monoxide, methane, and hydrogen are formed, together with acids and dark-coloured solids of great complexity similar to those occurring in the gelatinous deposit formed in transformers. A. R. Powell.

Examination of some transformer oils kept for long periods in the dark. H. von der Heyden and K. Typke (Petroleum, 1927, **23**, 15).—The increase in the tar-forming values of a number of transformer oils which have been kept in the dark for periods of one year or longer have been determined. Most of the oils were kept in metal containers, but some were in glass bottles; the vessels were not completely filled. The majority of the oils, and in particular one American non-sludging oil, show considerable deterioration. Of a number of Russian oils examined, those which had been subjected to the least refining showed the least deterioration on keeping. A. B. Manning.

Volumetric determination of aromatic hexahydro-hydrocarbons in petroleum and tar distillates. N. Dănăila and V. Stoenescu (Bul. Soc. Română Stiinte, 1926, **29**, 23—41).—The olefinic hydrocarbons are first determined in the manner previously described (B., 1924, 857; 1925, 835). The washed and dried residue is then dehydrogenated at 300—310° in presence of platinised asbestos (Zelinsky and Pawlow, B., 1923, 797). The unsaturated hydrocarbons are determined in the product as above, and again after a second dehydrogenation, and so on, until the amount of unsaturated hydrocarbons is constant. The saturated aromatic hydrocarbons are then determined. From these quantities the amount of aromatic hexahydro-hydrocarbons is calculated. S. K. Tweedy.

Moore ignition meter. W. R. Ormandy and E. C. Craven (J. Inst. Petrol. Tech., 1926, **12**, 650—654).—The meter used was similar to that described by Moore (cf. B., 1917, 109; 1924, 700), except that the explosion chamber had a slightly larger volume. Gas heating was used for moderate temperatures, and the block was heated to high temperatures by means of a nichrome wire winding. The spontaneous ignition temperature (S.I.T.) of heptane, previously given as 245°, has since been found to be 253°, great care being taken to remove all traces of hydrogen from the oxygen. Although successive readings of the S.I.T. can be obtained to within 1°, slight alterations of conditions may have an appreciable effect. No certain effect upon the S.I.T. was obtained by focussing the crater of an arc into the explosion chamber; by bringing the arc within a few inches of the chamber cover; by raising the potential of the block to 200 volts or by other attempts at ionisation; or by intensive drying of the liquid and the oxygen. The substitution of vapour for liquid raised the S.I.T. by about 5°. The S.I.T. are given of a number of compounds, and their significance is discussed. L. A. Coles.

Determination of benzene in alcohol solution. Ormandy and Craven.—See III.

Patents.

Coal washeries. A. Robinson, and Simon-Carves, Ltd. (E.P. 261,490, 1.9.25).—In coal-washing processes such as described in E.P. 22,189/13 (B., 1914, 410) and E.P. 110,994 (B., 1917, 1266), the slurry passes from the settling tank to a washing box of the air-pulsation or Baum type, the heavier and dirtier slimes are removed, and the lighter constituents of the slurry pass for drainage to inclined fixed and horizontal vibrating sieves. A. C. Monkhouse.

Manufacture of fuel agglomerates and their binders. F. M. Crossman (E.P. 244,053, 12.9.25. Conv., 5.12.24).—A binder is preferably made by mixing 100 lb. of starch with cold water and adding 1 lb. of sodium nitrate or other oxidising agent. The solution

is heated so as to gelatinise the starch, and 500 lb. of sulphite cellulose liquor (*d* 1·26) are added to enhance the cohesive properties of the binder. To the boiling mixture 400 lb. of oil residuum are added as a waterproofing agent. The binder thus prepared is admixed with powdered coal, preferably of low volatile content, in the proportion of 4—6% of binder. The agglomerate is pressed to shape and dried at 100—200°.

S. Pexton.

Coke manufacture. R. F. F. Fabry (E.P. 259,869, 8.6.26).—Coal is preheated to 100° by means of waste products of combustion and charged by a centrifugal fan into a small insulated service bunker. The preheating reduces the subsequent time of carbonisation, diminishes the water content of the distillation gases, and dispenses with coal-stamping and charging machines; a larger, denser, and harder coke is obtained.

A. C. Monkhouse.

Coking and distillation of carbonaceous materials. J. F. L. Moeller (E.P. 260,020, 25.6.25).—The material is subjected to a preliminary drying and cleaning, if necessary, and then preheated by hot air or by waste gases cooled to the desired temperature with air. The retort used may be of any type, and is supplied with steam superheated to a temperature at or above that of the fuel, viz., 260—400° for peat and 450—600° for coal. The steam is generated at high pressure and, after superheating, fed to the retorts at low pressure, the retorts being maintained at a pressure slightly lower than atmospheric. For stationary retorts a channel covered with perforated plates is used, the holes being protected by flaps. For rotary retorts a perforated central tube is used from which hollow perforated arms project which act also as stirrers.

A. C. Monkhouse.

Carbonising processes. W. E. Trent (E.P. 261,954, 8.2.26).—Pressed mixtures of coal and fuel oil of desired shape are fed into trays and slowly conveyed through a chamber for preliminary heat treatment at a low temperature (230—300°) in an atmosphere containing oxygen. The heating is conducted by hot elements temporarily fixed in each tray and on which the briquettes rest. The heating elements are replaced by other hot elements after each passage through the carbonising chamber. The briquettes, case-hardened by this treatment, are conveyed to a second retort and carbonised at 600—650° in an atmosphere free from oxygen. The product is of the same shape and size as the original briquettes.

S. Pexton.

Carbonising or cracking fuels. International Combustion Engineering Corp., Assees. of W. Runge (E.P. 253,499, 18.5.26. Conv., 12.6.25).—Finely divided fuel, *e.g.*, pulverised coal or atomised oil, is sprayed into a vertical retort, heated internally by ascending gases. The top of the retort is cooled to below 150° either by a water jacket or by cold gases entering above the carbonising zone; the hydrocarbons of higher b.p. are condensed and run back into the lower portion of the retort maintained at 570°, where they are carbonised.

A. C. Monkhouse.

Treating carbonaceous materials. W. E. Trent (E.P. 259,795, 3.12.25).—Dry pulverised coal or such coal in oil suspension, after mixing with a fluxing agent, *e.g.*, calcium or iron oxides or sodium carbonate, is carried in suspension in a stream of oil or air into a combustion chamber. Distillation takes place without fusion of the carbon particles. Carbonisation proceed, at 450° in a coil heated by gases passing down a refractory chamber in its centre. The products, oil, coke, and slags are separated by gravity, or water may be injected and the resulting emulsion subsequently treated.

A. C. Monkhouse.

Production of liquid fuel mixtures. T. H. Butler, F. J. W. Popham, J. C. Mann, and H. W. Robinson (E.P. 261,907, 17.11.25).—Coal tar pitch is blended with petroleum fuel oil by heating the former above its m.p. and gradually adding the oil during continuous agitation of the mixture. The fuel is burnt while still hot. The exact temperature at which mixing must take place is found by laboratory experiment; too low a temperature leads to a dull grainy mixture owing to separation of bitumen.

W. N. Hoyte.

Burning powdered or liquid fuel in furnaces. E. S. Suffern (E.P. 261,807, 29.5.25).—The fuel and "primary air" (about half that quantity required for complete combustion) are projected into the primary combustion chamber where the stream is deflected and diffused. Without extinction of the flame the stream passes to the secondary chamber where admixture with further air causes complete combustion. The burning fuel in the primary chamber is subjected to the radiant heat from the secondary chamber.

W. N. Hoyte.

Gas producers. A. Winkler (E.P. 247,565, 10.2.26. Conv., 11.2.25).—Steam or atomised water is sprayed into the fuel bed of gas producers through passages in the walls above the grate. The tubes for injection are continuous and arranged in recesses protected by firebrick at such a height above the grate as to prevent the formation of arches of clinker and the adherence of clinker to the walls. Various applications are described, in some of which the slope of the producer grate is adjustable, and an automatic extractor is provided at the end of the grate for the removal of ash and clinker.

A. C. Monkhouse.

Distilling or coking bituminous substances. Kohlenveredlung Ges.m.b.H., and C. Geissen (E.P. 261,290, 27.5.26).—In horizontal or vertical retorts in which a thin moving layer of fuel is carbonised by heat applied at one side, a wall is provided at the side of or above the fuel on the opposite side to the heating zone. This wall contains a number of channels to enable the products of distillation to be rapidly removed from the hot zone, the wall acting as an insulating medium preventing decomposition of the gases. A. C. Monkhouse.

Apparatus for the distillation of granulated and like combustibles. H. Wiedemann (E.P. 261,156, 9.10.25).—The retort used is of a beehive type enclosed in a furnace of similar shape heated at 400—600° by gas burners. The material is fed on to the bottom flat circular plate of the retort by means of a shoot with a flange to prevent the material passing to the coke conveyor. The charge is stirred by a vertical shaft fitted with flanged stirring arms which conveys the material round to the coke conveyor situate at the opposite

side to the gas offtake. The coke finally passes from the conveyor to a generator of inverted pear shape containing a similarly shaped core, fitted with spur-like projections, which can be rotated. Air and steam for the generator are supplied through channels in the casing walls. A. C. MONKHOUSE.

Combined process for carbonising coal and cracking oil. W. E. TRENT (E.P. 262,302, 15.2.26).—Pulverised coal, passing 100 mesh, mixed with water, is fed to an agglomerater, where it is mixed with petroleum residuum in the proportion of about 5 pts. by weight of coal to 1 pt. of hydrocarbon. The soft agglomerate is dried, formed into briquette shapes, and passed through a baking oven maintained at about 350° by hot flue gases, where the briquettes harden; they are then discharged. The vapours evolved from the oven pass to a compressor where they are compressed to about 100 lb./sq. in.; their temperature thereby rises to about 450°, cracking takes place, and the compressed vapours and liquid pass to a heat interchanger and thence to a pressure distillate storage. Fresh petroleum is also fed to the heat interchanger, the vapours evolved from the heating of this petroleum also pass to the above-mentioned compressor for cracking, and the unvaporised portion is stored and fed as required to the agglomerator. The compressor is actuated by steam, the flue gases from the boiler are utilised as a source of heat in the baking oven. W. N. HOYTE.

Converting methane gas into hydrocarbons of higher carbon content. LE PÉTROLE SYNTHETIQUE SOC. ANON., and A. FOLLIET (E.P. 261,267, 29.4.26).—Methane is slowly raised to 950° by passage between two refractory walls, 1 mm. or more apart, arranged by having a central core of refractory material inside a hollow tube. The gaseous products are then suddenly cooled to atmospheric temperature by means of a water cooler. The apparatus is maintained under a reduced pressure of 20 mm. of mercury by a pump. Using a natural gas containing 80% CH_4, 6% H_2, 2·5% CO_2, 2·6% C_3H_8 etc., 80% of the methane is converted into ethylene and olefines without the formation of naphthalene and tar. A. C. MONKHOUSE.

Complete gasification of bituminous fuels in alternately-operated generators. H. STRACHE (U.S.P. 1,611,842, 21.12.26. Appl., 6.8.21. Conv., 28.6.16).—See E.P. 117,083; B., 1920, 439.

Destructive distillation of vinasses. G. P. GUIGNARD (U.S.P. 1,609,712, 7.12.26. Appl. 6.11.24. Conv., 9.11.23).—See G.P. 421,787; B., 1926, 304.

Method and apparatus for cracking and distilling oils. MOTOR FUEL CORP., Assees. of C. F. RICHEY and P. Y. DUFFEE (E.P. 235,564, 16.6.24. Conv., 9.6.25).—See U.S.P. 1,530,091; B., 1925, 492.

Apparatus for treating hydrocarbon oils. J. P. PERSCH, Assr. to M. E. PERSCH (U.S.P. 1,611,669, 21.12.26. Appl., 22.11.20).—See E.P. 181,034; B., 1922, 580.

Apparatus for petroleum refining. C. JEFFERSON, Assr. to GRISCOM-RUSSELL Co. (U.S.P. 1,609,822, 7.12.26. Appl., 25.1.21).—See E.P. 174,569; B., 1923, 440.

Process and apparatus for converting high-boiling oils or hydrocarbons into stable low-boiling oils or hydrocarbons. D. L. THOMAS (U.S.P. 1,611,615, 21.12.26. Appl., 24.4.19. Renewed 15.5.26).—See U.S.P. 1,585,573; B., 1926, 623.

Charging horizontal gas retorts. F. G. MATTHEWS and J. G. W. ALDRIDGE (E.P. 262,625, 29.1.26).

Gas burners. RADIATION LTD., H. J. YATES, and M. HOWLETT & Co. LTD. (E.P. 262,659, 4.6.26).

Producing ammonium sulphate [from coal gas] (E.P. 262,320).—See VII.

Viscosity meter for testing the viscosity of liquids [lubricating oils] (E.P. 262,652).—See I.

III.—TAR AND TAR PRODUCTS.

Safety precautions in tar distillation.—RIEHM (Z. angew. Chem., 1927, **39**, 1597—1599).—An account of the precautions usually observed against risks of fire and explosion in the distillation of tar from pot-stills. C. IRWIN.

Determination of benzene in alcohol solution. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petrol. Tech., 1926, **12**, 636—649).—A rapid method has been devised for the determination of benzene in solution in alcohol of 95% strength and upwards, accurate to about 2% of the benzene present. A mixture of 50 c.c. of the solution with 5 c.c. of petrol, previously shaken with sulphuric acid to remove aromatic compounds, is shaken first with 150 c.c., then with 25 c.c. of 20% sodium chloride solution or with water, the aqueous layer being run off in each case. The percentage of benzene in the alcohol is approximately proportional to the difference between the refractive index of the residual hydrocarbons and those from a blank test using alcohol free from benzene. Preliminary notes are given on the application of the method for the determination of petrol in alcohol, and methods are discussed for the determination of the water content of alcohol–benzene mixtures. L. A. COLES.

Removal of tar from producer and coke-oven gas by electrostatic precipitation. WEYL.—See II.

Volumetric determination of aromatic hexahydro-hydrocarbons in tar distillates. DĂNĂILA and STOENESCU.—See II.

PATENTS.

Manufacture of asphalt or like substances in finely-divided condition. N.V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ, and F. R. MOSER (E.P. 245,418, 26.10.25. Conv., 2.1.25).—A stable "asphalt gel" proof against further coagulation is prepared by adding finely-divided substances to an emulsion of asphalt, tar, or pitch. These substances may be coagulating agents or, alternatively, an electrolyte is added simultaneously with the finely-divided material. Thus 1000 kg. of petroleum asphalt are emulsified so that the emulsion contains 50% of water; it is then mixed with 5 kg. of caustic soda and stirred into 1000 litres of a solution containing 5 kg. of crystalline aluminium chloride. Coagulation takes place, and the viscous gel is easily separated from excess water. The asphalt thus prepared is homogeneous and finely divided, and can be stored without further coagulation. S. PEXTON.

Production of liquid fuel mixtures (E.P. 261,907).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Microscopical tests for certain naphthalenesulphonic acids. W. Garner (J. Soc. Dyers and Col., 1927, 43, 12—13).—Two drops of a 40% solution of sulphate or chloride of zinc, magnesium, nickel, copper, iron, or manganese are mixed on a microscope slide with 3—4 drops of a 50% solution of the sulphonic acid, the solvent is allowed to evaporate, and the resulting crystalline mass examined. Characteristic structures are given for Schäffer's, naphthionic, Koch, and 2S acids, whilst S, H, and J acids yield no useful results.

B. P. Ridge.

Sensitisation of photographic plates. Leszynski. —See XXI.

Patents.

Azo dyestuffs. British Dyestuffs Corp., Ltd., M. Mendoza, and K. H. Saunders (E.P. 262,243, 13.11.25).—The substituted 2 : 4-diaminodiphenylsulphones obtained from salicylic acid (or its homologues or substituted derivatives) according to E.P. 245,865 (B., 1926, 233), especially 2 : 4-diamino-4′-hydroxydiphenylsulphone-5′-carboxylic acid and its 3′-methyl- and 3′-chloro-derivatives, may be used as end components in the manufacture of dyestuffs. Examples are given of the preparation of the following derivatives of the first-mentioned sulphone: *benzenesulphonic acid*-4-*azo*-2 : 4-*diamino*-4′-*hydroxydiphenylsulphone*-5′-*carboxylic acid*; the *benzenesulphonic acid*-3-*azo-compound*; the *naphthalene*-1-*sulphonic acid*-4-*azo-compound*; the 3-*nitrobenzenesulphonic acid*-1-*azo-compound*; the *benzenesulphonic acid*-2 : 5-*di*-(*azo-compound*); and the *diphenyl*-3 : 3′-*disulphonic acid*-4 : 4′-*di*-(*azo-compound*). The dyestuffs deepen from yellow towards blue with increasing complexity. The shades yielded on wool from an acid bath change on chroming, becoming at the same time fast to milling etc. Printed on cotton with a chrome mordant they yield shades fast to warm soap.

B. Fullman.

Polyazo dyes and process of making same. E. F. Hitch and F. H. Smith, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,610,946, 14.12.26. Appl., 10.10.23).—Two mols. of tetrazotised benzidine are coupled with 1 mol. of 1 : 8-aminonaphthol-3 : 6-disulphonic acid, the product is then coupled with 1 mol. of another component, and the remaining amino-group converted into a stable inorganic group. A. J. Hall.

Manufacture of dinitrotoluene. British Dyestuffs Corp., Ltd., E. H. Rodd, and R. W. Everatt (E.P. 263,018, 12.5.26).—Mixtures of dinitrotoluenes which remain liquid below 20° are prepared by nitrating mixtures of nitrotoluenes containing less than 45% of the *m*-compound, crystallising the liquid product at a suitable temperature, and removing the solid matter. *E.g.*, 685 pts. of nitrotoluene containing 33% *m*-, 32% *p*-, and 35% *o*-compound are nitrated during 3—4 hrs. at 40—50° with a mixed acid composed of H_2O 5·7%, HNO_3 24·3%, and H_2SO_4 70%. After stirring for 3 hrs. longer at 55—60°, the dinitrotoluene is separated in the usual way, washed free from acid, and dried by heating at 80° for a few hours. It is then crystallised by stirring at 18—20° for 24 hrs. The product is filtered or centrifuged, and thus yields 380—430 pts. of an oil of setting-point below 0° and 450—490 pts. of a solid of setting-point 54—60°. If the crystallisation is carried out at 25° for 24 hrs., 500 pts. of an oil of setting-point 18—20° are obtained, and the solid melts at 56—58°. (Cf. E.P. 17,128/13; B., 1914, 890.) A. Davidson.

Manufacture of nitro-derivatives of aromatic amines. J. W. Leitch & Co., Ltd., and A. E. Everest (E.P. 261,459, 13.8.25).—Dinitro-derivatives of *o*- and *p*-toluidines, *p*-chloroaniline, and aniline are prepared by treatment of the arylsulphonyl derivatives of these amines, or their mononitro-derivatives, with dilute nitric acid in excess, followed by hydrolysis of the products with sulphuric acid. *E.g.*, 70 g. of water, 26·1 g. of *p*-toluenesulpho-*o*-toluidide, and 25·2 g. of nitric acid (100% = 4 mols.) are mixed and heated slowly, with stirring, to 85°. The reaction which occurs further raises the temperature, which is then kept at 100° for several hours, the final product being a greenish-yellow flocculent mass. After cooling, this is filtered off and washed. By heating this with sulphuric acid (2 pts.) followed by dilution with water, *p*-toluenesulphonic acid is split off and 3 : 5-dinitro-*o*-toluidine is obtained.

A. Davidson.

Making aniline and other arylamines. W. J. Hale and J. W. Britton, Assrs. to Dow Chemical Co. (U.S.P. 1,607,824, 23.11.26. Appl., 5.2.25).—Aryl halides react with ammonia in the presence of a copper compound and metallic copper. B. Fullman.

Producing nitriles of the benzanthrone series. Kalle & Co., A.-G. (E.P. 243,026, 17.11.25. Conv., 17.11.24).—Halogenated benzanthrones are acted on by cuprous cyanide. *E.g.*, a mixture of 8 pts. of bromobenzanthrone (E.P. 20,837/06; B., 1907, 756) and 2·6 pts. of cuprous cyanide is heated at about 200—210° for some hours. The powdered melt is extracted with hot nitrobenzene, from which, on cooling, *cyanobenzanthrone*, m.p. 243—244°, crystallises. Fusion of the nitrile with caustic alkali or sodamide produces dark bluish-violet vat dyes. A. Davidson.

Manufacture of vat dyestuffs of the *iso*dibenzanthrone series. Badische Anilin- u. Soda-Fabr. (E.P. 242,620, 26.10.25. Conv., 7.11.24).—Benzanthrone-3-thio-ethers (alkyl or aryl, the latter including anthraquinonyl, benzanthronyl, and the like) containing a free 4-position are acted on by alkaline condensing agents in the presence or absence of inert diluents. *E.g.*, to a melt of 80 pts. of potassium hydroxide and 80 pts. of alcohol, previously heated to 135°, while distilling off excess of alcohol, 20 pts. of benzanthrone-3-*p*-thiocresyl ether are added while keeping the temperature at 135—140°. After stirring for 1 hr. more at 140—145°, the melt is diluted with water, and air is passed to precipitate the dye, which is *iso*dibenzanthrone practically free from dibenzanthrone. The same product can also be obtained from benzanthronyl-3 : 3′-sulphide. A. Davidson.

Manufacture of *iso*dibenzanthrones. J. Y. Johnson. From Badische Anilin u. Soda Fabr. (E.P. [a] 261,888 and [b] 262,030, 9.10.25).—(a) *Bz*-Halogeno-benzanthrones containing a free 4-position are converted

at comparatively low temperatures into *iso*dibenzanthrones by the use of mixtures of caustic alkalis and alkali alkoxides in presence of inert diluents. Instead of alcohols, substances such as benzaldehyde capable of forming alcohols by the action of caustic alkali may be used. Air should be excluded during the condensation. *E.g.*, 30 pts. of *iso*propyl alcohol are added to a stirred mixture at 10—15° of 200 pts. of benzene, 70 pts. of potassium hydroxide, and 10 pts. of pure *Bz*-chlorobenzanthrone, air being excluded. The benzene and alcohol are expelled by steam and the residue is vatted at the boil with hyposulphite. The vat is filtered and the dye precipitated by a current of air. (B) As condensing agents for the same purpose, metal compounds of aromatic amines in presence of an inert diluent may also be used. *E.g.*, to a solution at 15° of 8 pts. of sodium in 400 pts. of aniline are added all at once 40 pts. of chlorobenzanthrone (E.P. 20,837/06; B., 1907, 756). The temperature rises to about 25°, and stirring is continued at this point for 1—2 hrs. The dye is isolated, *e.g.*, by pouring the mass into dilute acid and filtering. It dyes vegetable fibres reddish-violet. The sodium may be replaced by other metals such as calcium, magnesium, or potassium. *Bz*-halogenobenzanthrones containing, in addition, halogen atoms in other positions may also be used, such as the dichlorobenzanthrones made by chlorinating (in the *Bz*-nucleus) 8- and 10-chlorobenzanthrones.

A. Davidson.

Intermediates, dyes, and dyeing. J. E. G. Harris, B. Wylam, J. Thomas, and Scottish Dyes, Ltd. (E.P. 261,139, 1.5.25).—The process of E.P. 247,787, 251,491, and 260,638 (B., 1926, 403, 625; 1927, 39) is extended to quinones in general, so as to yield quinone derivatives soluble in water, and capable of conversion into the original quinones by treatment with acid oxidising agents. When applied in particular to a number of anthraquinone derivatives which have hitherto been regarded as intermediates for, but not in themselves, dyestuffs, the derivatives so obtained may be used for dyeing or impregnating fibres, on which by treatment with acid oxidising agents the parent anthraquinone derivatives are then regenerated. Examples are given of the preparation of derivatives from anthraquinone, 1:4-di-*p*-tolylaminoanthraquinone, 1-methylaminoanthraquinone, 1:4-dimethyldiaminoanthraquinone, etc.

A. Davidson.

Manufacture of intermediate products [2:3-hydroxynaphthoic arylides] suitable for the preparation of azo dyestuffs. British Synthetics, Ltd., and E. B. Higgins (E.P. 262,958, 28.12.25).—The reaction between 2:3-hydroxynaphthoyl chloride and an aromatic amine containing no electronegative substituent can be made practically quantitative as regards both amine and chloride by vigorously mixing the two substances in presence of water and a slight excess of weak alkali. The mixing of the components may be done in varying order, and the acid chloride may be used in solid form or in solution in an indifferent solvent. *E.g.*, to a paste of 93 g. of aniline with 100 g. of sodium bicarbonate and 50 g. of water a suspension or solution of 206·5 g. of 2:3-hydroxynaphthoyl chloride in 200 g. of benzene is slowly added with efficient stirring or grinding, the temperature not exceeding 60°. After stirring for ½ hr. more, 100 g. of water are slowly added and the benzene is driven off by steam. The mass is then neutralised to phenolphthalein by adding hydrochloric acid, and the anilide washed and dried. The yield is 97% or more. Alternatively, the chloride may be added to a mixture of the amine and alkali, followed by water, or a mixture of the chloride and the amine is added to the alkali and water. A. Davidson.

Production of vat dyestuffs. D. G. Rogers, Assr. to Nat. Aniline & Chemical Co., Inc. (U.S.P. 1,609,965, 7.12.26. Appl., 17.6.21. Cf. G.P. 331,283; B., 1921, 294).—*N*-Dihydro-1:2′:2:1′-anthraquinoneazine is prepared by gradually adding about 30 kg. of potassium hydroxide to a mixture of about 200 litres of mineral oil and 40 kg. of β-aminoanthraquinone at about 220—225°, raising the temperature to about 230°, and allowing the water vapour evolved to escape. A. Davidson.

Dibenzanthronyl product. Dyestuffs and intermediates. R. F. Thomson and J. Thomas, Assrs. to Scottish Dyes, Ltd. (U.S.P. 1,607,491—2, 16.11.26. Appl., 20.2.26. Conv., 21.10.24).—See E.P. 251,313; B., 1926, 576.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Amidated cotton. P. Karrer and W. Wehrli (Z. angew. Chem., 1926, **39**, 1509—1514).—Cotton and other cellulose materials, when treated with ammonia, yield a fibre containing 1—3% N, the reaction either being the substitution of an amino-group for a hydroxyl group, or partial oxidation of the cellulose, and such amidated yarns are dyed directly with acid dyes. A so-called "immune" yarn is prepared by the partial esterification of cotton with *p*-toluenesulphochloride, it has considerable immunity to substantive dyestuffs, and can be treated with ammonia to form amidated yarn. Instead of ammonia, organic basic nitrogen compounds can be used for the treatment of cotton or "immune" yarns; *e.g.*, aliphatic amines such as ethylamine or dimethylamine in aqueous solution give a yarn which can be dyed with tartrazine, cyanol, etc. Benzylamine gives a yarn with strong affinity for acid dyes; molten urea, phenylhydrazine, piperidine, and also tertiary amines are applicable. The essential quality of amidated yarns is their smooth dyeing power with acid dyes, which is not shared by "immune" or ordinary cotton. A list of suitable dyestuffs is given. W. G. Carey.

Changes in the tenacity and elongation of artificial silk in the normal and wet conditions. II. Y. Kami (Cellulose Ind., Tokyo, 1926, 2, 39—40).—The loss of tenacity of artificial silk is greater in boiling water than in water at normal temperature, and the loss increases as the time of boiling is prolonged. This increased loss is partly attributable to the expansion of occluded gas and the consequent disruption of the cohesive forces of the cellulose molecules; the mechanical disturbance due to ebullition also plays a part. The elongation, with a few exceptions, is diminished in the same manner. Drying the boiled silk at a high temperature has an injurious effect on tenacity and elongation, but if the material be dried out in the air, not above 35°, the physical characteristics are substantially restored to the original

value. Under these conditions, boiling for less than 30 min. has no effect on the tenacity and elongation, but longer boiling, *e.g.*, for 1 hr., produces a permanent deterioration in the physical properties, which may be likened to elastic fatigue. J. F. Briggs.

Viscose. VIII. G. Kita, R. Tomihisa, and K. Sakurada (Cellulose Ind., Tokyo, 1926, **2**, 40—46).—The influence of the conditions of preparation of the viscose on the tenacity and elongation of the films has been studied. The ageing of the alkali-cellulose (up to 10 days) has no appreciable influence on the tenacity of the films even though the viscosity is thereby greatly reduced. Longer ageing of the alkali-cellulose, however, retards the rate of coagulation of the film. Under-ripe viscose gives rough opaque films on coagulation which subsequently become clear; fully-ripe viscose gives clear films immediately. Viscose prepared from well-aged alkali-cellulose or dissolved in more concentrated alkali does not undergo such marked changes in viscosity during ripening as viscose prepared under opposite conditions. The tenacity of the films, however, is unaffected in either case, and with few exceptions the tenacity tends to increase with duration of ripening in an asymptotic manner until it arrives at a constant value; the elongation coefficient of the films passes through a maximum point with duration of ripening. The concentration of alkali in the viscose has no very definite influence on the tenacity, but the elongation is very notably increased when the xanthate is dissolved in 8—12% sodium hydroxide solution. Dilution of the viscose has no effect on the tenacity of the film. J. F. Briggs.

Action of sodium hydroxide on cotton cellulose. W. F. A. Ermen and S. H. Jenkins (J. Soc. Dyers and Col., 1927, **43**, 9—12).—The absorption of sodium hydroxide by well-bleached Egyptian single-cotton yarn at the ordinary temperature, as determined by a weighing and titration method, shows that there is no evidence for chemical combination when solutions of concentrations up to *d* 1·175 and beyond *d* 1·250 are used. Between these points, however, a constant absorption value indicates the formation of a compound of the composition $C_6H_{10}O_5,0\cdot6NaOH$. The rate of absorption from a solution of *d* 1·250 increases extremely rapidly for the first 30 sec., but afterwards only slowly for times up to 30 min. B. P. Ridge.

Rate of the disincrusting reaction in the cooking of wood by the soda process. A. Ruby (Pulp and Paper Mag., 1926, **24**, 1421—1424).—A method, based on the observation that the isolation of cellulose in the soda process appears to follow the laws of unimolecular reactions, is described for calculating, with the aid of the data obtained from two cooks made under different known conditions of time, concentration of liquor, and temperature, the effects of various combinations of these factors, so that the optimum cooking conditions may be found without the necessity for a long series of experimental cooks. D. J. Norman.

Patents.

Manufacture of parchmentised or vulcanised fibre. R. Arnot (E.P. 260,374, 17.8.25).—Parchmentised or vulcanised fibre is rendered non-corrosive by treatment at any stage of the process, *i.e.*, before, during, or after vulcanisation, with a medium which is capable of forming with the hydrolysing agent a non-corrosive compound. For example, cellulosic material may be pre-treated with latex or a drying oil, such as tung oil, or the vulcanised board, after removal of as much of the zinc chloride as is practicable, may be treated with a solution of an alkali, lead acetate, a salt of silicic or phosphoric acid, an oleate, an albuminate, an organic base, tung oil, or the like. The medium may be chosen to impart special characters such as flexibility or resistance to water or fire, or may provide an adhesive for uniting several layers into one solid block by, for example, causing the precipitation of sticky zinc soaps on the surface of the board, as is the case when certain alkali soaps are used. D. J. Norman.

Obtaining fibres from flax, hemp, jute, ramie, and other suitable plants. J. J. Feuge and L. R. Browne (E.P. 239,495, 6.7.25. Conv., 6.9.24).—The material is fed from a hopper vertically between fluted rollers, the motion of which causes folding of the stalks and pressing together of the folds, thus breaking away extraneous matter. The cleaned fibres are then straightened. B. P. Ridge.

Process for chemically varying vegetable fibres. Heberlein & Co., A.-G. (E.P. 255,453, 13.7.26. Conv., 14.7.25).—Fibres containing cellulose are treated with a halogen compound of phosphorus (such as the oxychloride) in the presence of an alcoholic solution of sodium hydroxide. Cotton so treated undergoes substantially no shrinkage, acquires an increased affinity for basic dyestuffs, and loses its affinity for substantive dyes, so that white effects may be obtained by dyeing with substantive dyes fabrics containing treated and untreated material. B. P. Ridge.

Manufacture of artificial silk or the like. J. O. Zdanowich (E.P. 260,642, 6.5.25).—Freshly-coagulated artificial silk threads, particularly cellulose acetate threads, are wound on a resilient bobbin or spool, for example, a gapped cylinder or a cylinder made in the form of a spiral, to prevent the development of undue stresses during the drying of the thread, and at the same time to provide means whereby the initial tension of winding is automatically increased slowly and continuously as the drying proceeds. D. J. Norman.

Manufacture of artificial filaments, film bands, and the like from viscose. H. Hawlik (E.P. 242,242, 8.9.25. Conv., 31.10.24).—In order to cause the precipitation of large quantities of sulphur both inside and on the surface of viscose filaments, alkaline or ammonium sulphides or polysulphides, either alone or containing in solution metal sulphides, *e.g.*, of arsenic, antimony, or tin, which are precipitable in an insoluble form by the acid in the spinning bath, are added to the viscose solution, or used for dissolving the cellulose xanthate. A suitable spinning solution contains 8% of cellulose and 6—10% of sodium sulphide, or 7% of titratable alkali and 0·2—1% of antimony sulphide. D. J. Norman.

Manufacture of derivatives of cellulose and formic acid, and of dyed or undyed materials therefrom. Fabr. van Chem. Producten, and A. ter Horst (E.P. 260,650, 30.6. and 22.7.25).—Cellulose formate of high

formic acid content suitable for the manufacture of artificial filaments is made by treating cellulosic material, including modified, regenerated, and partly esterified celluloses, with concentrated formic acid (95% or stronger) at temperatures between 0° and 5°, in the presence of a catalyst such as phosphorus tri- or penta-chloride, chlorosulphonic acid, sulphuric acid, or zinc chloride. Dissolution of the cellulose requires 24—40 hrs., and the resulting solution is stable at the ordinary temperature if the catalyst is rendered inactive by the addition, for example, of a salt of formic acid. *Example*: 50 kg. of dried wood cellulose are treated at 0° with 520 kg. of 95% formic acid and 4·5% of sulphuryl chloride. After 48 hrs. a clear viscous solution is obtained to which are added, while stirring, 80 kg. of sodium formate and sufficient formic acid to produce a spinning solution of the required viscosity; organic liquids or aqueous salt solutions, *e.g.*, sodium formate solution, may be used as coagulants. The resulting filaments have, even after partial saponification, a high tensile strength, their breaking length exceeding 15,000 metres in the dry state and 10,000 metres in the wet state. Formate silk may be dyed by any of the methods used for dyeing cellulose acetate. D. J. Norman.

Manufacture of wood pulp. I.-G. Farbenind. A.-G. (E.P. 262,693, 17.9.26).—A continuous bisulphite boiling process is carried out with a lye containing about 1·25% CaO (or CaO and MgO) and about 5·3% total SO_2 (corresponding to about 2·4% free, 2·9% available, and 1·45% combined SO_2), so that when the bisulphite has disappeared about 2% free SO_2 still remains, and is not removed while the boiling is terminated. A mixture of calcium and magnesium bisulphites gives better results than calcium bisulphite alone. B. P. Ridge.

Pulping raw cellulosic material. G. A. Richter, Assr. to Brown Co. (U.S.P. 1,610,323, 14.12.26. Appl., 24.12.25).—Fibre is liberated from raw cellulosic material by means of an acid cooking liquor containing nitre cake in solution. B. P. Ridge.

Manufacture of wood pulp. E. R. Barker and F. J. Dooley (U.S.P. 1,612,484, 28.12.26. Appl., 15.5.26).—The excess bisulphite liquor and sulphur dioxide from one digester are transferred to a second at a point substantially removed from its top and bottom. B. P. Ridge.

Producing pulp high in resistant cellulose. G. A. Richter, Assr. to Brown Co. (U.S.P. 1,602,553, 12.10.26. Appl., 11.11.24).—Sulphite pulp is treated with the black liquor resulting from the alkaline digestion of wood. D. J. Norman.

Defibration of paper and the like. O. C. Winestock (E.P. 262,270, 10.12.25).—Waste-paper stock, after a preliminary treatment in a shredder, is diluted to a consistency of 5—7% with warm water, preferably containing a suitable detergent, and fed into a defibreing apparatus provided internally with two parallel shafts which are driven in the same direction at 2500—4000 r.p.m., and carry a series of propellers so placed that those on the one shaft interleave with those on the other. The blades on each propeller are alternately of opposite pitch, and are not designed to forward the stock through the machine, this being effected, and the process rendered continuous, by the hydrostatic head of the mixture in the feeding hopper. D. J. Norman.

Pre-treating wood chips in the production of soda cellulose. E. L. Rinman (U.S.P. 1,609,832, 7.12.26. Appl., 21.4.20. Conv., 16.8.19).—See G.P. 369,606; B., 1923, 447.

Preventing easily-inflammable articles from inflaming, and extinguishing fire. A. Eichengrün (U.S.P. 1,612,104, 28.12.26. Appl., 3.8.23. Conv., 10.8.22).—See G.P. 390,840; B., 1924, 506.

Treatment of cellulose hydrate. C. F. Cross (U.S.P. 1,611,001, 14.12.26. Appl., 4.5.25. Conv., 12.5.24.)—See E.P. 236,336; B., 1925, 707.

Hydro extractors [for laundries]. Baker Perkins, Ltd., and F. Dewhurst (E.P. 262,954, 22.12.25).

Spinning box for use in the artificial silk industry. Soieries de Strasbourg Soc. Anon., and E. Bronnert (E.P. 262,874, 21.9.25).

Dyeing of acetylcellulose (E.P. 261,822).—See VI.

Dyeing or colouring of yarns etc. [and cellulose acetate silk] (E.P. 262,506).—See VI.

Paper insulation for electric cables etc. (E.P. 251,931).—See XI.

Treatment of water (E.P. 262,369).—See XXIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Identity of digallic acid with tannin as mordants for basic dyes. P. P. Victorov (J. Soc. Dyers and Col., 1927, 43, 12—17).—Synthetic digallic acid has the same characteristic properties as the natural tannins. It gives a blue coloration with ferric chloride, and forms precipitates with gelatin, quinine acetate, tartar emetic, and basic dyes, those with the dyes being soluble in excess of the acid. It is absorbed by cotton, and acts as a mordant in basic dyeing after fixation with tartar emetic. In mordanting cotton, the tannin molecule decomposes, and only the digallic acid acts in forming the tannin-antimony complex and dyeing the material, so that for this purpose the synthetic acid may replace the natural tannin. B. P. Ridge.

[Dyeing] amidated cotton. Karrer and Wehrli.—See V.

Patents.

Production of fast dyeings on the fibre. A. G. Bloxam. From A.-G. für Anilin-Fabr. (E.P. 262,537, 17.9.25).—Textile fibres are dyed by impregnation with a compound (capable of coupling with a diazo compound) and a nitrite and afterwards treating with an acidic solution of a diazotisable amine. Alternatively, particularly for purposes of printing, the diazotisable amine may be applied to the fibre before treatment with the acid solution. A reddish-orange shade on cotton yarn is obtained by impregnating 10 g. of yarn with 200 c.c. of a solution containing per litre 10·5 g. of the anilide of β-hydroxynaphthoic acid, 17·5 c.c. of caustic soda (*d* 1·31), 22 c.c. of Turkey-red oil, 8 c.c. of 30% formaldehyde, and 30 g. of sodium nitrite, and then treating it with 200 c.c. of a solution containing per litre 0·95 g. of the hydrochloride of 2 : 5-dichloroaniline and 7·5 g. of aluminium sulphate. A. J. Hall.

Production of uni- or multi-coloured effects [on textiles etc.]. Brit. Dyestuffs Corp., Ltd., H. Jackson, and D. Carter (E.P. 262,601, 18.12.25).—Solutions of nitrocellulose, in the form of celluloid, in organic solvents and coloured by the addition of inorganic pigments, or organic dyes or lakes prepared therefrom, are poured on to water, whereby a very thin coloured film is formed on the surface, and textile materials, feathers, leather, paper, rubber, cork, etc. are then drawn through the water and afterwards dried to remove the organic solvent, the coloured film adhering to the material being treated. The resulting shades are fast to water and to moderate soaping. A satisfactory solution for producing a coloured film consists of 9 pts. of celluloid film scrap, 200 pts. of amyl acetate, 100 pts. of acetone, 15 pts. of benzene, and 1 pt. of Rhodyle 4 G or other dye. A. J. Hall.

Methods and apparatus for dyeing, coating, or colouring [textile] materials or other articles. Two-Tone Corp., Assees. of P. Mijer (E.P. 236,539, 22.6.25. Conv., 5.7.24).—Textile fabric is dyed by passage through a cloud consisting of an atomised dye solution or coating liquor (cf. E.P. 236,194; B., 1926, 976), the fabric being simultaneously treated so that the deposited dye or coating substance is dried on the fabric before emerging from the cloud. A. J. Hall.

Methods and apparatus for treating textile materials. Two-Tone Corp., Assees. of P. Mijer (E.P. 237,230, 23.6.25. Conv., 16.7.24).—Textile fabrics are coloured, dyed, or coated with dye liquids by passing the material, held stretched in a tenter mechanism, through a chamber in which a cloud of finely divided particles of dye liquid is produced by an atomiser, arrangements having been made to remove any drops of liquid formed in the atomisation. Diagrams of the apparatus used are given. (See preceding abstract.) A. Davidson.

Dyeing of acetylcellulose. J. W. Leitch & Co., Ltd., and A. E. Everest (E.P. 261,822, 11.8.25 and 11.5.26).—Acetylcellulose is dyed by dinitro-derivatives of aromatic amines in which both nitro-groups are in the *o*-position to the amino-group, such as 3 : 5-dinitro-*p*-toluidine and 2 : 6-dinitro-*p*-chloroaniline. *E.g.*, to the dye-bath filled with cold water 5 pts. of a 20% paste of finely divided 3 : 5-dinitro-*p*-toluidine are added, the goods entered cold and, after raising slowly to 50°, worked at this temperature for $\frac{1}{2}$—$\frac{3}{4}$ hr., then rinsed and dried. No additions to the dye-bath are necessary, nor does the nitro-compound require emulsification. Since the dyeings may be carried out in a neutral, acid, soap, or alkaline bath, other colours can be used in combination to produce compound shades. A. Davidson.

Dyeing or colouring of yarns and fabrics or other materials [containing cellulose acetate silk]. Brit. Celanese, Ltd., and G. H. Ellis (E.P. 262,506, 7.9.25).—Cellulose acetate silk materials are dyed or printed with vat or sulphur dyes, hydrolysis of the silk being avoided by the use of the sodium or potassium salts of hydroxy- or polyhydroxy-derivatives of benzene, naphthalene, and similar compounds, instead of the caustic soda usually employed. Suitable salts include sodium or potassium salts of phenol, cresol, xylenol, pyrocatechol, resorcinol, guaiacol, α- and β-naphthol. A. J. Hall.

Production of fast greenish-yellow shades on acetyl silk. J. Baddiley and J. Hill, Assrs. to British Dyestuffs Corp., Ltd. (U.S.P. 1,611,986, 28.12.26. Appl., 20.5.25. Conv., 20.6.24).—See E.P. 236,037; B., 1925, 708.

Squeezing and guide rollers etc. for yarn dyeing machines. Brit. Cotton and Wool Dyers' Assoc., Ltd., E. A. Barker, and F. Ellison (E.P. 262,909, 21.11.25).

Intermediates, dyes, and dyeing (E.P. 261,139).—See IV.

Manufacture of derivatives of cellulose and formic acid, and of dyed or undyed materials therefrom (E.P. 260,650).—See V.

Varying [the behaviour of] vegetable fibres [towards dyestuffs] (E.P. 255,453).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Evolution of iron sulphide in contact with water. S. Veil (Compt. rend., 1926, **183**, 1286—1288).—The oxide obtained by heating iron sulphide prepared by precipitation of a ferrous or ferric salt with ammonium sulphide, and afterwards treated with hot water, is more magnetic than the corresponding oxide derived from iron sulphide prepared in the cold. The sulphides show the same difference, the former being most stable to oxidation, and the latter having a magnetisation of the same order as that of its corresponding oxide. The sulphides prepared in the warm and cold are designated the "modified" and "ordinary" sulphides, respectively, and the phenomena are analogous to those obtained for iron hydroxide, the "ordinary" and "modified" forms of this compound showing the same differences in properties as the corresponding sulphides, and yielding the same kinds of oxides (cf. A., 1924, ii, 557). On redissolving the oxide and reprecipitating the sulphide successively, the magnetisations of both rise to a maximum and then fall. J. Grant.

Recovery of alumina from natural silicates. H. Kassler (Chem.-Ztg., 1926, **50**, 917).—The aluminium content of natural silicates such as clay or kaolin may be rendered soluble in sulphuric acid by heating the silicate under pressure with sodium carbonate solution. The alkali first decomposes the silicate with the formation of a sodium aluminosilicate and carbon dioxide. On cooling, still under pressure, the sodium carbonate is regenerated and the alumina and silica are liberated in a granular hydrated form from which the former is readily dissolved by treatment with dilute sulphuric acid, leaving a mass of almost pure amorphous silica. Practically the whole of the sodium carbonate used is recovered for use again in the process, but from time to time it must be purified from organic matter by evaporation and calcination. Most of the potash in the silicate follows the alumina into the sulphuric acid solution, and, when the alumina is removed by addition of ammonia under conditions adapted to restrain the precipitation of

iron, a mixed ammonium potassium sulphate may be obtained by evaporation, which is of value as a fertiliser.
A. R. POWELL.

Alkaline treatment of leucite. F. JOURDAN (Chim. et Ind., 1926, **16**, 902—907).—A mixture of 100 pts. of finely-powdered leucite with 130 pts. of well-burnt lime and 400 pts. of water, when heated under pressure for some hours in an autoclave fitted with a stirrer, yields a potash lye containing 95% of the potassium, the residue containing all the aluminium. To obtain the aluminium as well as the potassium a mixture of granular leucite and limestone, with iron oxide as a flux, is melted in an electric furnace at about 1000°, and the product cooled very slowly. The whole mass falls to powder, and is leached and filtered. Carbon dioxide is passed through the potassium aluminate solution, giving a 90% yield of potassium carbonate and leaving 80% of the aluminium as alumina free from iron and containing 0·1—0·2% of silica. If finely-powdered leucite and lime be heated for some time at 650—700° with a substance capable of combining with the potassium at that temperature, stable potassium salts are obtained. *E.g.*, the addition of chromium oxide gives a good yield of potassium chromate.
W. G. CAREY.

Extraction of bauxite by caustic soda without pressure. B. NEUMANN and O. REINSCH (Z. angew. Chem., 1926, **39**, 1542—1545).—Investigation of the proportion of alumina extracted from French bauxite by caustic soda solution showed that it varied directly with the temperature and also with the concentration of the sodium hydroxide. The use of sodium hydroxide in proportions in excess of those practically employed in the Bayer process, viz., 1·8 mols. of Na_2O to 1 mol. of Al_2O_3, increases the percentage extraction somewhat, but is not admissible as it interferes with the subsequent precipitation of aluminium hydroxide. The working conditions of the Bayer process, a pressure of 4—6 atm. and a temperature of 170°, are therefore the readiest means of attaining the temperature necessary, which is the essential point. A solution of sodium hydroxide boiling at 170° under atmospheric pressure is equally efficient, but is too concentrated to handle in practice. Neither is it possible to raise the b.p. of a more dilute solution sufficiently by adding sodium chloride.
C. IRWIN.

Technical analysis of burnt magnesite. O. KALLAUNER (Chem. Listy, 1926, **20**, 549—553).—For the determination of silica in high-grade burnt magnesite ore evaporation with hydrochloric acid is usually sufficient. The separation of lime from magnesia is preferably effected by precipitation with ammonium oxalate from the solution slightly acidified with acetic acid, using either a small volume of liquid with the addition of ammonium chloride, or a volume of 500 c.c. without ammonium chloride. The separation by sulphuric acid or lithium sulphate gives slightly high results. Moisture is determined by heating for 1—2 hrs. at 105° and loss on ignition by heating for 15 min. over the full heat of a Teclu burner.
A. R. POWELL.

Volatile barium sulphate. F. L. HAHN (Chem.-Ztg., 1926, **50**, 934; cf. Krauss, A., 1926, 368).—The fact that barium sulphate moistened with sulphuric acid colours the flame of a bunsen burner green, whereas when moistened with hydrochloric acid only a faint coloration is produced, is not necessarily due to the volatility of barium sulphate, but may be caused by the formation of a spray of very finely-divided barium sulphate formed by the rapid decomposition of barium hydrogen sulphate or the pyrosulphate. The green colour of the flame obtained by igniting the vapours evolved from a heated mixture of barium nitrate, strong sulphuric acid, and methyl alcohol is probably due to methyl nitrate, as similar colorations are obtained by substituting strontium or ammonium nitrate for the barium salt.
A. R. POWELL.

Analysis of rock-salt. W. TOELDTE (Chem.-Ztg., 1926, **50**, 933—934).—From the results of 94 analyses of rock-salt it is shown that the magnesium content occurs chiefly as sulphate and not as chloride, and that calcium is always present as sulphate. In calculating the results of an analysis, therefore, the calcium found should be reckoned as sulphate, any excess of sulphuric acid being calculated to magnesium sulphate. If there is still an excess of sulphuric acid this is calculated to sodium sulphate; if an excess of magnesium it is calculated to magnesium chloride.
A. R. POWELL.

Preparation of sulphur dioxide from gypsum. B. NEUMANN (Z. angew. Chem., 1926, **39**, 1537—1542).—If the necessary amount of gypsum is added to blast-furnace slag, the reaction $CaS + 3CaSO_4 = 4CaO + 4SO_2$ occurs, and after heating the mixture at 1400° for 15 min. a yield of over 90% of sulphur dioxide is obtained. If the slag contains no calcium sulphide, a similar decomposition of the gypsum occurs at this temperature. A blast furnace producing 200 tons of slag per day could decompose 20 tons of anhydrite per day (equal to 14 tons of sulphuric acid) with a consumption of 20,000 cub. m. of blast-furnace gas for temperature maintenance. Calcium sulphide alone is rapidly oxidised by dry air, even at temperatures below 1000°, but only one-third of the sulphur is obtained as sulphur dioxide, the remainder being fixed as calcium sulphate. A mixture of sulphide and sulphate gives similarly poor yields (at 800—1100°) in a current of air; in nitrogen, however, desulphurisation is complete at 1100°. Similar results are obtained with slags containing calcium sulphide and sulphate, and the procedure of blowing slag with air for sulphur recovery is considered fallacious.
C. IRWIN.

Analysis of silicates. LASSIEUR.—See VIII.

Plaster of Paris. CHASSEVENT.—See IX.

Preparation and effectiveness of basic copper sulphates for fungicidal purposes. HOLLAND, DUNBAR, and GILLIGAN.—See XVI.

Production of concentrated acetic acid. SUIDA.—See XX.

PATENTS.

Manufacture of phosphoric acid. H. BLUMENBERG, JUN., Assr. to STOCKHOLDERS SYNDICATE (U.S.P. 1,609,239, 30.11.26. Appl., 30.4.25).—Phosphoric acid is prepared by treating finely ground calcium phosphate in the presence of water with sulphur dioxide under pressure.

The sulphur dioxide is in excess of the amount required to form sulphurous acid with the water. W. Clark.

Treatment of zinc oxide [for rubber compounding]. New Jersey Zinc Co., Assees. of F. G. Breyer, E. H. Bunce, and J. H. Weikel (E.P. 253,875, 17.5.26. Conv., 18.6.25).—Zinc oxide produced from roasted sulphide ore is reduced in acidity, and thereby made suitable for rubber compounding, by treatment at 100—150° with steam. The oxide is showered through a steam-heated zone or conveyed through a tunnel or furnace having a heated zone filled with steam, and is dried by passing through a tube heated with combustion gases. W. G. Carey.

Production of aluminium oxide. Höganäs-Billesholms Aktiebolag (E.P. 252,693, 27.4.26. Conv., 27.5.25).—Bauxite is melted in an electric furnace with sufficient carbon to reduce the silicic acid and iron oxide; at the last stage of the process more carbon is added, which forms aluminium carbide, and this on cooling contains the principal part of the impurities. To obtain a still purer product iron oxide with carbon for its reduction is added to the melt; the iron is precipitated as a spray and carries to the bottom nearly all the metallic impurities. The molten oxide is tapped off, granulated in water, and after pulverising is treated with sulphuric acid to decompose the aluminium carbide and to eliminate the last traces of metallic impurities. W. G. Carey.

Manufacture of stannic acid or oxides of tin. H. Harris. From K. B. Heberlein (E.P. 263,034, 11.6.26).—Impure stannic acid which may have been obtained from the treatment of impure molten lead with sodium hydroxide (cf. B., 1924, 522) is purified from arsenic and antimony by heating in solid form at 1000° with sufficient sodium or potassium carbonate to remove the impurities, the product being ground with water and leached. Any further impurities in the residue after filtration are removed by repeating the process. W. G. Carey.

Purifying plumbiferous chloride liquors. Consortium für Nassmetallurgie (E.P. 249,465, 24.8.25. Conv., 20.3.25. Addn. to E. P. 240,401; B., 1925, 990).—The prior process for purifying plumbiferous ores etc. is hastened if, after treatment with calcium carbonate and oxidation, sufficient alkaline substance is added to precipitate all the iron, but only about 1% of the lead, followed by sodium sulphide sufficient to precipitate metals electro-negative to lead, but leaving all but about 0·5% of the lead in solution. The extract is freed from the combined precipitates, and is converted into any desired lead compound. W. G. Carey.

Recovery of ingredients of waste lead battery paste. A. Stewart, Assr. to C. L. Constant Co. (U.S.P. 1,610,899, 14.12.26. Appl., 10.10.25).—The waste paste is fused with an acid salt of an alkali metal. H. Royal-Dawson.

Production of anhydrous hyposulphites. L. K. Riggs, Assr. to E. R. Squibb and Sons (U.S.P. 1,609,773, 7.12.26. Appl., 23.12.24).—Crystalline hyposulphite is heated in an atmosphere of water vapour to a temperature sufficiently high to drive off water of crystallisation, and the anhydrous product maintained at that temperature to prevent recombination of the water during its removal along with water vapour. J. S. G. Thomas.

Production of sodium sulphide. H. Freeman, Assr. to Canada Carbide Co. (U.S.P. 1,609,615, 7.12.26. Appl., 12.6.25).—Sodium sulphate is reduced by melting with a carbonaceous reducing agent and a flux capable of maintaining about four times its weight of sodium sulphide fluid at 1000—1500°. J. S. G. Thomas.

Producing ammonium sulphate [from coal gas]. Soc. Anon. des Fours à Coke Semet-Solvay & Piette (E.P. 262,320, 7.4.26. Conv., 22.3.26).—Coal gas, freed from ammonia but still containing acid impurities, is treated with aqueous ammonia, and the resulting solution is brought into contact with a suspension of finely-pulverised calcium sulphate. The insoluble materials (calcium carborate and excess sulphate) are separated after each treatment, and the solution is heated to liberate the volatile acids and ammonia, which is cooled and used again in the first part of the process. H. Royal-Dawson.

Obtaining sulphur from alkaline-earth sulphates. Salzwerk Heilbronn A.-G., T. Lichtenberger, and K. Flor (E.P. 258,828, 9.2.26. Addn. to 251,942, 29.1.26).—The conversion of alkaline-earth sulphides into the chlorides is accelerated by adding magnesium chloride to the melt of the alkali chloride and alkaline-earth sulphate before or after the reduction with coke. Hydrogen sulphide, the evolution of which is increased by passing steam into the melt with the elimination of magnesium oxide, is worked up for sulphur, while the alkaline-earth chlorides evolved from the sulphides and insoluble in the melt are soluble in the molten alkali chlorides. H. Royal-Dawson.

Recovery of nitrogen oxides. E. M. Jones, G. E. Beavers, A. M. Fairlie, and J. N. Houser, Assrs. to Tennessee Copper & Chemical Corp. (U.S.P. 1,610,288, 14.12.26. Appl., 1.8.24).—The separation of sulphur dioxide and nitrogen oxides from a mixture with other gases is effected by absorption in activated carbon. H. Royal-Dawson.

Manufacture of fluorine. W. J. Tennant. From N. V. Philips' Gloeilampenfabr. (E.P. 262,918, 28.11.25).—Oxyfluorides of titanium, zirconium, or hafnium, prepared by evaporating a solution of the fluorides and drying *in vacuo* at 300°, are heated at a temperature above 200°, either with a peroxide, or in the presence of oxygen which has been dried by sulphuric acid and liquid air. W. G. Carey.

Separating hydrogen cyanide, ammonia, and carbon dioxide from gas mixtures. O. Liebknecht, Assr. to Roessler & Hasslacher Chem. Co. (U.S.P. 1,606,767, 16.11.26. Appl., 8.11.24. Conv., 26.11.23; cf. E.P. 26,259/98; B., 1899, 1020).—Gas mixtures containing hydrogen cyanide, ammonia, and carbon dioxide are passed into a solution or suspension of an alkaline-earth or magnesium salt, *e.g.*, calcium sulphate, calcium chloride, or magnesium chloride, which is more soluble than the corresponding carbonate. The hydrogen cyanide is unaffected, and if, for example, calcium sulphate is employed, calcium carbonate is precipitated, and ammonium sulphate remains in solution. On heating this latter mixture ammonia and carbon dioxide

are volatilised, with reprecipitation of calcium sulphate. If the original gas mixture contains a stoicheiometric excess of ammonia over carbon dioxide, an excess of the latter is produced by adding flue gases, and the mixture of carbon dioxide and hydrogen cyanide obtained from the calcium sulphate treatment is passed into a mixture of water, alkali sulphate, and lime, when a solution of alkali cyanide and a precipitate of calcium carbonate and sulphate are obtained. T. S. WHEELER.

Synthesis of ammonia. G. CLAUDE, Assr. to LAZOTE, INC. (U.S.P. 1,605,647, 2.11.26. Appl., 18.11.21). —See E.P. 150,744; B., 1922, 99.

Art of producing ammonia synthetically. E. COLLETT, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,608,926, 30.11.26. Appl., 17.8.23. Conv., 17.8.22).—See E.P. 237,394; B., 1925, 757.

Catalyst for the synthesis of ammonia. C. URFER (U.S.P. 1,605,875, 2.11.26. Appl., 29.5.23. Conv., 12.6.22).—See E.P. 199,025; B., 1924, 256.

Production of ferric oxide. O. S. NEILL (U.S.P. 1,607,206, 16.11.26. Appl., 14.11.25. Conv., 24.10.24). —See E.P. 251,310; B., 1926, 584.

Manufacture of calcium arsenate. J. ALTWEGG, Assr. to SOC. CHIM. DES USINES DU RHÔNE (U.S.P. 1,608,288, 23.11.26. Appl., 16.6.23. Conv., 27.4.23).—See E.P. 214,951; B., 1925, 70.

Method of producing alkali-metal xanthates. L. ROSENSTEIN, Assr. to GREAT WESTERN ELECTRO-CHEMICAL Co. (Re-issue 16,474, 16.11.26, of U.S.P. 1,507,089, 2.9.24).—See B., 1924, 908.

Method for producing synthetic precious stones. O. RUFF (U.S.P. 1,605,073, 2.11.26. Appl., 2.2.23. Conv., 15.3.22).—See G.P. 395,419; B., 1924, 747.

Method of producing hydrogen. F. G. LILJENROTH and M. LARSSON, Assrs. to PHOSPHORUS-HYDROGEN Co. (U.S.P. 1,605,960, 9.11.26. Appl., 18.9.23. Conv., 29.5.23).—See G.P. 409,344; B., 1925, 448.

Separation of one or more constituents of liquid mixtures, [Concentration of acetic acid.] W. BADER, Assr. to AMERICAN CELLULOSE AND CHEMICAL MANUF. Co., LTD. (U.S.P. 1,609,393, 7.12.26. Appl., 6.7.23. Conv., 6.9.22).—See E.P. 204,196; B., 1923, 1160.

Production of hydrocyanic acid. G. BREDIG and E. ELÖD, Assrs. to R. KOEPP & Co. (U.S.P. 1,610,035, 7.12.26. Appl., 21.11.24. Conv., 7.12.22).—See E.P. 229,774; B., 1925, 315.

Lime hydrator. H. MISCAMPBELL (U.S.P. 1,610,572, 14.12.26. Appl., 15.11.24).—See E.P. 257,793; B., 1926, 946.

Production of tungsten and molybdenum carbide in lumps of various sizes. H. LOHMANN (U.S.P. 1,610,061, 7.12.26. Appl., 26.8.21. Conv., 16.4.14).—See G.P. 286,184; B., 1915, 1250.

Apparatus for salt purification. J. E. NASH, Assr. to WORCESTER SALT Co. (U.S.P. 1,610,307, 14.12.26. Appl., 17.2.25).—See U.S.P. 1,549,019; B., 1925, 757.

Production of nitrogen-hydrogen mixture for the synthetic production of ammonia. F. W. DE JAHN, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,610,076, 7.12.26. Appl., 21.9.23).—See E.P. 222,147; B., 1925, 757.

VIII.—GLASS; CERAMICS.

Influence of oxide admixtures on the properties of silica bricks. K. ENDELL and R. HARR (Stahl u. Eisen, 1926, **46**, 1870—1876).—Addition of up to 6% of lime, ferric oxide, or alumina to the usual raw material for the manufacture of silica bricks invariably increases the sp. gr., but has no appreciable action on the subsequent growth of the brick during firing. Alumina tends to retard the conversion of quartz into tridymite, whilst lime and, to a much greater extent, ferric oxide assist the conversion. The ferric oxide does not react with the silica, but a small amount is converted into calcium ferrite by the lime used as a bond, and the remainder is present as magnetite after firing. In the presence of iron oxide the tridymite and cristobalite effects occur between 100° and 300°, but no quartz effect at 575° is noticeable in the fired bricks; hence they are relatively more resistant to rapid changes of temperature than those containing lime or alumina. The porosity of silica bricks remains unchanged with increase of lime to 8%, increases considerably with a rise in the alumina content, but decreases on the addition of ferric oxide to about 17 vol. % with 6% Fe_2O_3. Alumina decreases the crushing strength of silica bricks and lowers the softening point under a constant load of 1 kg./cm.^{2}, by 125° for 2% Al_2O_3 and by 275° for 6% Al_2O_3. Up to 6% CaO has no effect on the crushing strength or softening point of the bricks, whilst 6% Fe_2O_3 increases the former considerably and decreases the latter by only 20°. For practical purposes, therefore, silica bricks should not contain more than 1·5% Al_2O_3, but may contain up to 8% CaO with safety. The presence of 4—6% Fe_2O_3 is an advantage when the bricks are to be used in an oxidising atmosphere, but in a reducing atmosphere there is danger of the formation of fusible ferrous silicates. A. R. POWELL.

Analysis of silicates. A. LASSIEUR (Bull. Soc. chim., 1926, [iv], **39**, 1759—1766).—The silicate is treated with hydrochloric acid either before or after fusion with sodium carbonate, evaporated to dryness, and heated at 105—110° till no more fumes of acid are evolved. By treating cold with 5 c.c. of conc. hydrochloric acid and, after 10 min., adding 100 c.c. of boiling water the silica can be filtered. Contrary to Treadwell's statement ("Quantitative Analysis"), silica is precipitated completely and in a pure state under these conditions. The hot solution is now just neutralised with ammonia, when the hydroxides of iron, aluminium, and titanium are precipitated, their separation being effected by calcining and treating with potassium bisulphate. The precipitate is obtained free from alkalis, alkaline-earths, and magnesium, but excess of ammonia must be avoided as it precipitates magnesium and also dissolves appreciably the aluminium hydroxide. Both these effects can be avoided by adding ammonium salts, but some 10 g.-mol. are required per g.-mol. of aluminium. Calcium is precipitated by adding saturated ammonium oxalate solution to the hot ammoniacal solution; the presence of the great excess of alkali salts from the fusion is not deleterious, but magnesium is carried down and can be separated by reprecipitating the calcium with ammonium oxalate in a solution of p_H 4 or 5. In the final precipitation of magnesium as the ammonium

phosphate, excess ammonia as usually stated is unnecessary, a p_H slightly over 7 being sufficient.
S. J. Gregg.

Patents.

Manufacture of a fused quartz product. E. Thomson, Assr. to General Electric Co. (U.S.P. 1,610,182, 7.12.26. Appl., 28.12.23).—Crystalline silica is mixed with a small proportion of a more fusible vitreous material, moulded, and heated to a temperature sufficiently high to soften the binder, but insufficient to fuse the silica. B. W. Clarke.

Glass composition. W. C. Taylor, Assr. to Corning Glass Works (U.S.P. 1,609,329, 7.12.26. Appl., 10.10.23).—A glass substantially free from silica and containing boric oxide, alumina, an oxide of a second group element, and not more than 10% of alkali of which more than 50% is potash. H. Holmes.

Producing glass suited particularly for insulating purposes. Patent-Treuhand Ges. für Elektr. Glühlampen m.b.H. (E.P. 256,189, 11.6.26. Conv., 1.8.25).—A glass for insulating purposes contains silica, potassium oxide, and one or more oxides of bi- or ter-valent metals, and is prepared from a batch containing no reducing agents, but which contains iron oxide as an auxiliary flux, and is melted in an oxidising atmosphere. A. Cousen.

Glass containing germanium. L. M. Dennis (U.S.P. 1,607,817, 23.11.26. Appl., 14.1.25).—A transparent glass is obtained by fusing together a compound of a basic element with a germanium compound, which forms the acidic constituent of the glass. (Cf. B., 1927, 42.) A. Cousen.

Process and apparatus for annealing sheet glass. A. E. White. From Pittsburgh Plate Glass Co. (E.P. 262,629, 16.2.26).—A continuously formed sheet or ribbon of glass is fed into a gas-heated lehr chamber at a temperature above 560°, and heat is applied along its side edges as it cools down to and through the critical annealing range, additional heat being applied to the centre of the sheet as it approaches this range to prevent warping and cracking. B. W. Clarke.

Electric furnaces [for glass]. J. K. B. Raeder, and Aktieselskabet Raeders Elektroglasovn (E.P. 262,535, 17.9.25).—Solid electrodes wholly submerged in a bath of molten glass which acts as a resistance to the electric current are provided with a surface sufficiently large to reduce the current density and to ensure that the electrodes are outside the actual heating zone of the bath, thereby improving the appearance of the glass produced. B. W. Clarke.

Manufacture of refractory articles of pure zirconium oxide. Deutsche Gasglühlicht-Auer-Ges.m.b.H. (E.P. 246,480, 21.1.26. Conv., 23.1.25).—Pure zirconium oxide obtained from the basic sulphate can be made into refractory articles capable of withstanding temperatures up to about 2600° without shrinkage or cracking by sintering the oxide at above 1800°, working up the articles without particularly fine grinding, and burning in two stages, first at about 1300° and subsequently above 2000°. If the mass of zirconium oxide is subjected to pressure the temperature of burning may be lowered to 1600°.
W. G. Carey.

[Silica] brick-making. T. C. Pond, Assr. to Johns-Manville Inc. (U.S.P. 1,612,649, 28.12.26. Appl., 25.2.26).—A mixture of comminuted sulphur and diatomaceous earth is moulded, dried, and fired, to produce a silica brick by the conversion of the sulphur into the gaseous state, and the fluxing and fritting of the silica particles with the alkaline earths associated therewith. B. W. Clarke.

Tunnel ovens. Dressler Tunnel Ovens, Ltd., and O. Vermorcken. (E.P. 262,678, 19.8.26).—In a tunnel oven of the Dressler type, the base and vertical side walls are constructed, in one with the oven structure, of ordinary refractory bricks, the chamber being surmounted by an ogival dome formed of curved plate made of thinner refractory material. B. W. Clarke.

Enamelling of cast and sheet metal. C. Totot-Gibaru (E.P. 262,159, 16.6.25).—A process for enamelling metal by the "dry" method consists in applying a mixture of silica 2—25%, zinc oxide 20—35%, hydrated borax 35—70%, alkali nitrate 2—20%, alkali fluorides 0—8%, and calcium carbonate 0—30%. If additional opacity is required, antimony oxide or stannic oxide is added. A decorative effect may be obtained by superposing on the above enamel, by the dry method, a mixture containing 5—15% SiO_2, 1—3% Al_2O_3, 10—20% CaO, 30—40% ZnO, 12—18% alkalis, 15—30% B_2O_3, 1—3% F, with oxide of iron, cobalt, or manganese to colour. A. Cousen.

Vitreous containers for electric lamps etc. (E.P. 262,498).—See XI.

IX.—BUILDING MATERIALS.

Plaster of Paris. M. L. Chassevent (Bull. Soc. d'Encour., 1926, **125**, 789—803).—The course of the setting of plaster of Paris was followed by observing the electrical conductivity of the solution which forms in the immediate vicinity of the plaster, and also by measurement of the heat change. Anhydrous calcium sulphate prepared below 300° is transformed immediately on contact with water into the semihydrate, forming a saturated solution which is supersaturated with respect to gypsum, the difference between the solubilities of the semihydrate and of gypsum being greater the lower the temperature. After a time gypsum crystallises out and heat is evolved; this corresponds with the commencement of the setting. If the anhydrous calcium sulphate is prepared above 300°, a different form is produced, which rehydrates very slowly, the time increasing with rise of temperature of preparation. A saturated solution of calcium sulphate semihydrate at 16° does not crystallise for 25—28 min. if care is taken to avoid inoculation, but if nuclei of gypsum are present in the original material the velocity of setting is increased. Setting is accelerated also by the presence of salts which increase the SO_4''-ion concentration, and by acids which do not cause a precipitate. Poor plaster work is generally due to an excess of water for setting; with increasing amounts of added water the apparent density and the resistance to compression of the plaster decrease.
E. S. Hedges.

Influence of oxide admixtures on properties of silica bricks. Endell and Harr.—See VIII.

Patents.

Manufacture of a building material. W. H. Barker, Assr. to R. J. D. Orn (U.S.P. 1,610,211, 7.12.26. Appl., 28.9.21).—Silica and asbestos fibre are digested under heat and pressure in the presence of an alkaline hydroxide. B. W. Clarke.

Method of making brick or other burned-clay products. P. Maynard (U.S.P. 1,609,416, 7.12.26. Appl., 8.3.26).—A mixture of clays is burnt at above 1100° to give a product having an iron oxide content from 2½ to 3½% and less than 25% of lime. B. W. Clarke.

Process of cementation in the ground. T. Blandford, A. Gee, and H. E. Potts (E.P. 262,223, 14.10.25).—Fissures in water-bearing strata are sealed by injecting a dilute solution of a caustic alkali or alkali carbonate, with or without the addition of an organic material, *e.g.*, soap, which preferably lowers the surface tension, followed by the injection of a cement suspension in the usual way. B. W. Clarke.

Manufacture of plaster. Lambert Frères & Cie. (E.P. 243,015, 13.11.25. Conv., 13.11.24).—Gypsum is calcined at 500—800° until the product is completely dehydrated and has *d* 2·70—2·80, forming a non-crystalline plaster which does not set, but may be added to ordinary plaster to reduce its speed of setting. B. W. Clarke.

Plaster for building or other purposes. J. E. Evans-Jackson. From G. H. Whittle (E.P. 262,195, 8.9.25).—Powdered gypsum is rapidly calcined and mixed with pulverised alum and rock nitre, forming a plaster with slow-setting properties. B. W. Clarke.

Dry [quick-setting] lime mixture. F. C. Mathers and S. C. Shirley, Assrs. to National Lime Assoc. (U.S.P. 1,607,472, 16.11.26. Appl., 5.11.25).—A substance which accelerates the setting of lime mixtures, such as magnesium carbonate, is made into a paste with a 1% solution of starch, gum arabic, or similar water-soluble substance, dried, ground, and mixed with lime to form a stable composition. T. S. Wheeler.

Manufacture of mortar-formers and mortar. J. H. Ditter (E.P. 262,232, 30.10.25).—A composition for use as a mortar consists of a mixture of magnesium compounds with alkali silicates in a colloidal form, with or without the addition of fluorides, fluosilicates, milk of lime, carbon tetrachloride, water-repelling materials, etc. The product is resistant to water and chemical action. B. W. Clarke.

Preparation of compositions for use as positive catalytic agents for hydraulic binding materials. C. Schneider (E.P. 262,294, 28.1.26).—Calcined calcium chloride, with or without the addition of calcium oxide and a retarder such as calcium sulphate, is mixed with sufficient gelatinous silicic acid (prepared by evaporating a solution of Portland cement or the like in hydrochloric acid) to form a solid product which is resistant to storage. B. W. Clarke.

Producing cellular or porous concrete. E. I. Lindman (E.P. 243,308, 9.6.25. Conv., 24.11.24).—A heat-insulating and hydraulising material, *e.g.*, slag or ashes of coal or coke, lava, etc., containing silicates but relatively little lime, and having a sp. gr. such that the material remains suspended throughout the wet mass, is used as aggregate in making porous concrete of the type comprising cement, water, and a metal powder adapted to generate gas. B. W. Clarke.

Manufacture of bricks, building blocks, slabs, and the like. F. Mulligan (E.P. 262,224, 14.10.25).—Hydraulic cement or plaster prepared from gypsum according to E.P. 238,949 (cf. B., 1925, 851) is mixed with aggregate, *e.g.*, sand or asbestos, and with colouring or finishing materials, and finally moulded. B. W. Clarke.

Artificial soapstone. F. Wynkoop (U.S.P. 1,606,490, 9.11.26. Appl., 23.4.25).—A mixture of soapstone with a cement of high alumina content, giving high tensile strength and low absorption. B. W. Clarke.

Process for obtaining pressed asphalt compositions and similar products. J. Jachzel (E.P. 248,752, 3.3.26. Conv., 3.3.25).—Limestone powder, sand, or the like is agitated with a dilute colloidal bitumen solution, and an electrolyte is added to cause spontaneous impregnation of the limestone and precipitation of an asphalt powder, which is separated, dried, and pressed. B. W. Clarke.

Water-repellent composition [cement]. H. S. Lukens, Assr. to Solidifier Corp. (U.S.P. 1,607,405, 16.11.26. Appl., 15.6.26).—An emulsion of paraffin wax, oil, or other water-resisting substance (1—3%) in water, when used in place of water in the manufacture of cements and other setting materials, yields products of increased resistance to the action of water. T. S. Wheeler.

Process of road treatment. P. G. Ekström (U.S.P. 1,606,928, 16.11.26. Appl., 6.7.25. Conv., 22.5.25).—A mixture for the treatment of roads consists of sulphite lye mixed with sufficient calcium hydroxide solution to convert all the organic acid compounds present into salts. A hard, asphalt-like surface is obtained. T. S. Wheeler.

Manufacture of asphalt or like substances in finely-divided condition (E.P. 245,418).—See III.

Silica brick (U.S.P. 1,612,649).—See VIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Influence of carbon and silicon on the graphitisation of white castings. P. Chevenard and A. Portevin (Compt. rend., 1926, 183, 1283—1284; cf. B., 1925, 504).—A three-dimensional diagram has been constructed to show the influence of carbon and silicon on the temperature of spontaneous graphitisation of a pure casting containing less than 0·1% of manganese, sulphur, or phosphorus. The proportions of carbon and silicon are such as to include most industrial castings, and hypoeutectics in the iron-carbon system. The curves for the effect of silicon are approximately hyperbolic, and indicate that beyond 2·5—3·5% Si the graphitisation temperature is almost constant at about 600°. The rectilinear graphitisation isotherms indicate that silicon and carbon together promote graphitisation, the effect

of the latter being most marked for silicon contents of less than 2%. J. Grant.

Desulphurising action of fluorides on molten iron. E. Wilke-Dörfurt and H. Buchholz. (Stahl u. Eisen, 1926, **46**, 1817—1820).—The sulphur content of an iron containing 3·35% C, 0·22% S, and 0·63% Mn is lowered by melting in contact with calcium or manganese fluoride, silicofluorides of sodium or manganese, or with sodium aluminium fluoride. In the presence of a slag this action is less marked, though the sulphur in the slag is also decreased. The results support previously advanced theories that the sulphur is removed by formation of fluorine compounds, and that the process does not involve migration of sulphur from iron to slag, but a removal of sulphur from the whole (iron and slag) system. L. M. Clark.

Determination of graphite and combined carbon in cast iron. W. A. Burford and W. Baader (Z. anal. Chem., 1926, **69**, 456—457).—1 g. of the sample is dissolved in 20 c.c. of nitric acid (*d* 1·2) in a 100 c.c. Nessler tube heated in a water bath. When the evolution of gas has ceased the tube is cooled and 25 c.c. of 30% sodium hydroxide solution are added to precipitate the iron as hydroxide, followed, after again cooling, by cold water up to the 100 c.c. mark. The precipitate is allowed to settle and 15 c.c. of the clear solution are then transferred to an Eggertz tube, in which its colour is compared with that of a similar solution prepared in the same way from a standard steel of known carbon content. The liquid and precipitate remaining in the Nessler tube are treated with an excess of nitric acid, and the graphite is collected and determined by the method of Ledebur. A. R. Powell.

Resistance of steel containing copper to atmospheric corrosion. K. Daeves (Stahl u. Eisen, 1926, **46**, 1857—1863).—A review of recent work on the effect of adding copper to steel to improve its resistance to corrosion; 0·2—0·25% Cu increases the life of steel constructions exposed to the atmosphere and to flue gases by at least 50% owing to the formation of a thin, smooth, dense, and tightly adherent coating of oxide quite distinct from the loosely adherent rust that forms on ordinary steel. The life of galvanised and tinned plates made with this steel is also very appreciably prolonged. A. R. Powell.

Dependence of electrical resistance of nickel steel on composition, temperature, and heat treatment. F. Ribbeck (Z. Physik, 1926, **39**, 787—812; cf. B., 1926, 949).—Many curves and tables are given showing variation of resistance with temperature from $-180°$ to 1000° for steels with varying nickel content. The irreversibility of steels with less than 30% Ni is attributed to supercooling; the α—γ change of irreversible nickel steels is hindered by rapid cooling and by increased carbon content. The transition temperatures are shown on the resistance–temperature curves, but for reversible steels the magnetometric method is more sensitive; if the steel contains more than 35·2% Ni there is no reversible change; chilling raises the temperature at which the α—γ transition takes place. E. B. Ludlam.

Corrosion. R. H. Ruthven (Gas J., 1926, **176**, 495—498).—The corrosion of iron is reviewed with special reference to the influence of impurities in the iron. Corrosion in a gas holder was traced to ammonium cyanide in the holder water. The impure water was replaced with town's water, and the holder was scraped and repainted; corrosion of the submerged plates was still not eliminated by this procedure, and a layer of paraffin oil was floated on the surface of the tank water. After a period of three years no further corrosion was detected. A number of failures in cast-iron water mains have been traced to the acid nature of the surrounding clay. One remedy is to remove the clay from direct contact with the pipe and replace it with chalk. S. Pexton.

Alloys resistant to corrosion and their properties. E. H. Schulz and W. Jenge (Z. Metallk., 1926, **18**, 377—386).—A general review of the properties of alloys resistant to corrosion and of their behaviour towards various corrosive media is given, followed by a more extensive survey of the iron–silicon and so-called "stainless" iron alloys. The resistance of iron–silicon alloys to 5% hydrochloric acid increases rapidly with rise of silicon content from 12 to 18%, but the brittleness also increases considerably. The stainless iron and steel alloys are all more or less attacked by hydrochloric acid and chloride solutions; Krupp's V2A steel (20% Cr, 7% Ni, and 0·25% C), however, is resistant to alkali salts in general, nitric acid, cold sulphuric acid of any strength, boiling phosphoric acid more dilute than 40%, acetic acid, and cold oxalic acid solutions, but is readily attacked by boiling 20% sulphuric, 80% phosphoric, oxalic, and tartaric acids. All the chromium–iron and steel alloys containing more than 12% Cr are resistant to oxidation up to 800°, and their tensile strength is decreased only slightly by heating to 500°. A. R. Powell.

Acid-resisting alloys with a nickel base. W. Rohn (Z. Metallk., 1926, **18**, 387—396).—The action of 10% solutions of nitric, sulphuric, hydrochloric, acetic, and phosphoric acids on iron, nickel, cobalt, copper, chromium, molybdenum, and tungsten, and on 35 alloys of these elements, has been investigated at 20° and at 100°. In general, the presence of manganese decreases the resistance of iron–chromium and nickel–chromium alloys towards hydrochloric and sulphuric acids, whereas a small amount of silicon usually improves the acid-resisting properties. Addition of carbon to chromium–iron alloys improves their resistance to hydrochloric acid, but decreases their resistance to sulphuric acid. Nickel is rather more beneficial than molybdenum in chromium–iron alloys, but molybdenum increases the resistance of nickel–chromium alloys towards hydrochloric acid; the most useful alloys contain about 60% Ni, 15% Cr, 2·5—7% Mo, remainder iron with a little manganese. These alloys are readily workable, and retain their mechanical properties to at least 500°. Water-bath rings of the alloy with 7% Mo retained their lustre after 100 days' exposure in a laboratory fume-cupboard. A. R. Powell.

Problem of acid-resisting metallic materials. W. Guertler (Z. Metallk., 1926, **18**, 365—376).—In a comprehensive review of the theoretical principles,

chemical, thermochemical, and electrochemical, underlying the problem of the production of an acid-resisting alloy for any desired purpose, the author discusses the construction of affinity series of the metals derived from their behaviour under various conditions, and the circumstances that may influence the position of a metal in these series, and points out the great effect of the formation of solid solutions in alloys on their behaviour towards corroding media. A. R. POWELL.

Behaviour of cold-rolled brass in tension and compression. G. MASING and W. MAUKSCH (Wiss. Veröff. Siemens-Konz., 1926, **5**, 142—155).—From theoretical considerations it is shown that, if no secondary effects occur, the curve showing the extension of brass against the load should be similar in shape to that obtained by plotting the compression against the load. Tests on cold-worked brass in extension and compression show that the compression curve deviates appreciably from the theoretical, and especially so just above the elastic limit. The amount of this deviation depends on the nature of the cold work, being more marked after deformation by extension than after deformation by compression, and greater after a low-temperature anneal (250°) than after a high-temperature anneal (625°). In all cases the load required to cause a standard degree of deformation within the plastic region is less than that theoretically required, and it is suggested that this is due to some property of the metal which causes a weakening of the resistance to compression after the metal has been subjected to plastic elongation. A. R. POWELL.

Change in the properties of tin bronzes caused by pressing and forging. A. SCHLEICHER (Z. Metallk., 1926, **18**, 322—323).—The mechanical properties of a bronze containing 92·71% Cu, 7·08% Sn, and 0·21% Ni were hardly affected by deformation in a hydraulic press to 34% of its original thickness or by forging to 37%, but the structure varied considerably from the top to the bottom of the ingot. Whereas at the top where the deforming force was applied the grains were of fairly even structure and regular in size, at the bottom they were much coarser and more irregular. In the case of the pressed bars many slip planes were visible, and the boundaries between the crystal grains were often perforated with small fissures; these defects were not nearly so marked in the forged bars. A. R. POWELL.

Viscosity [of copper, brass, and bronze] at high temperatures. J. COURNOT and R. PAGÈS (Rev. Mét., 1926, **23**, 701—708; cf. B., 1925, 994, and A., 1927, 13).—The viscosity–temperature curves for copper, brass, and aluminium bronze are roughly parallel to the corresponding breaking strain–temperature curves, but are considerably below them. The copper curve is approximately a straight line up to 400°. The curve for aluminium bronze (10% Al) is also a straight line, nearly perpendicular to the temperature axis up to 300°; at this point a sharp deflexion occurs, after which the curve is convex to the temperature axis up to 600°, then parallel to it. Above 300° the viscosity of brass is greater the higher the copper content, but is considerably decreased by addition of lead. The limit of viscosity of 80/20 brass is about 30·5 kg./mm.^{2} and the curve showing the elongation during the first hour under a load above the elastic limit rises to a maximum at a load of about 21 kg./mm.^{2} The curves obtained for time of rupture–load at 400° for 60/40 brass and at 500°, 600°, and 700° for 10% aluminium bronze are hyperbolic in character, and agree closely with those found by French and Tucker (A., 1927, 13). A. R. POWELL.

Electrical conductivity of technical aluminium. G. MASING and C. HAASE (Wiss. Veröff. Siemens-Konz., 1926, **5**, 183—192; cf. B., 1925, 675).—The precipitation from solid solution of the impurities in commercial aluminium brought about by heating at 300—350° is accelerated by previous cold work, but retarded by previous annealing at 500—600°. Addition of beryllium or magnesium has no effect on this behaviour, but iron accelerates and silicon retards the separation of the impurities. Very variable results for the electrical conductivity are obtained by ageing at 140—170° aluminium that has been quenched from various temperatures between 250° and 550° after hot or cold rolling, the highest value always being obtained, however, with metal quenched from 250—350°. Prolonged annealing at 170° of cold-rolled aluminium will produce the maximum conductivity while reducing only slightly the tensile strength and ductility. The presence of adsorbed gas affects the conductivity to a very small degree. A. R. POWELL.

Influence of pouring temperature and mould temperature on the properties of a lead-base anti-friction alloy. O. W. ELLIS (Fac. Appl. Sci., Toronto, 1926, Bull. 6, 143—159).—Compression tests on bearing metal alloys containing 80—85% Pb, 15—10% Sb, 3·5—5% Sn, and 1·5—0% Cu showed that replacement of lead by antimony increases the hardness and resistance to compression, and that replacement of tin by copper increases the resistance to compression without affecting the hardness. The mould temperature, *i.e.*, the rate of cooling, has a greater effect on the compression strength than has the pouring temperature; the higher the mould temperature the lower the stress at which plastic flow first occurs, and the smaller the percentage reduction in height at the time of rupture, although the stress required to cause rupture remains fairly constant. Addition of copper to a lead–tin–antimony bearing metal containing 80% Pb, 15% Sb, and 5% Sn prevents segregation in the cast ingot owing to an intermetallic reaction between the γ-cubes of SnSb and the copper with the formation of needles of Cu_2Sb. This reaction is accompanied by an evolution of heat in the liquid alloy at 334°. The compounds Cu_3Sn and Cu_2Sb can coexist in an alloy of the composition 91% Sn, 4·5% Sb, and 4·5% Cu, and needles in which the interior consists of purple Cu_2Sb and the outer layers of Cu_3Sn frequently occur in castings of this alloy. A. R. POWELL.

Effect of air on cupellation losses [of silver]. J. T. KING (Fac. Appl. Sci., Toronto, 1926, Bull. 6, 255—341).—In order to determine the effect of air on the loss of silver during cupellation numerous experiments were carried out in an electric muffle into which a pipe for admission of regulated air supplies was fixed in such a manner that the air could be directed to any part of the muffle. The action of the air supply is twofold; firstly, it cools the muffle, cupel, and litharge, and,

secondly, it increases the rate of oxidation and thereby raises the temperature of the alloy. When the air has its greatest cooling effect then the loss is lower the greater the air supply, but when it has its greatest heating effect the converse holds good; the actual conditions in the muffle are usually somewhere between these extremes. The loss of silver increases with the temperature of the surface litharge (which is always above that of the surrounding air), and is greatest towards the end of the cupellation; it is also increased by an increase in the rate of cupellation brought about by increasing either the volume or temperature of the air supply. The smaller the proportion of silver to lead in the alloy the greater the percentage loss of silver in the cupellation. Frozen buttons and the presence of litharge pools on the cupel during any part of the cupellation cause large but variable losses. The best results and lowest losses are obtained by using an independent air supply provided by an adjustable suction fan so arranged that the air is drawn in a uniform manner across the entire width of the muffle, and by so arranging the heat distribution from front to rear of the muffle that the cupelling buttons appear to be at the same temperature throughout. The cupels should be of the same dimensions, as deep as possible, closely packed in the furnace, and preferably covered by hoods or rings. A. R. POWELL.

Hollowing of tungsten bars by central fusion. P. WOOG (Bull. Soc. chim., 1926, [iv], **39**, 1708—1711).—In the preparation of tungsten for wire-drawing, very heavy currents are passed through bars of the metal in an atmosphere of hydrogen. Occasionally the m.p. (3655° Abs.) is accidentally exceeded, and the bar ruptures, when its interior is found to be hollow, and the metal immediately surrounding the hollow shows evidence of melting and subsequent recrystallisation. The cooling of the outside layers must be due both to radiation and to conduction and convection by the gas. Langmuir has shown that the conductivity of hydrogen is abnormally high at high temperatures, owing to dissociation into atoms, being proportional to the fifth power of the absolute temperature above 2600° Abs. To test this view, the tungsten was heated in an atmosphere of nitrogen, when 29% less heat was necessary to cause rupture; this tallies with Langmuir's figure 23% for the difference in heat conductivities between nitrogen and hydrogen at 3400° Abs. S. J. GREGG.

Effect of working on the physical properties of tungsten. J. W. AVERY and C. J. SMITHELLS (Proc. Physical Soc., 1926, **39**, 85—96).—Changes in the density and resistivity of tungsten during swaging and drawing have been measured. The densities of pure and thoriated tungsten (0·63% ThO_2) from the sintered bar down to wires 0·02 mm. diam. are given. The density rises rapidly during swaging to a maximum value within 0·5% of the density of the perfect tungsten crystal, and then falls slowly with further working. Measurements on specimens cleaned in successive stages by etching reveal the presence of low-density surface layers. The resistivity falls rapidly in the early stages of swaging, and reaches a minimum when the density is a maximum. It then increases at a rate which is nearly an exponential function of the diameter. C. J. SMITHELLS.

Deoxidation of nickel. G. MASING and L. KOCH (Wiss. Veröff. Siemens-Konz., 1926, **5**, 170—174).—The cause of the brittleness of commercial nickel is not the presence of oxide but the presence of sulphide. Nickel and nickel oxide form a eutectic (m.p. 1438°, 0·214% O) which is evenly distributed throughout the crystal grains, whereas the $Ni-Ni_3S_2$ eutectic (m.p. 625°) segregates along the grain boundaries, thus causing inter-crystalline fracture when the metal is rolled either hot or cold. Addition of manganese removes the oxygen, but results in the formation of manganese sulphide, which forms a eutectic with nickel (m.p. 1325°, 3% S), and this also segregates to the grain boundaries. A further addition of magnesium destroys this eutectic and removes nearly the whole of the sulphur as magnesium sulphide; what little remains suspended in the metal is evenly distributed as minute globules in the crystallites, and has no deleterious effect on the rolling properties.
A. R. POWELL.

Hardening of metals. R. S. ARCHER (Trans. Amer. Soc. Steel Treat., 1926, **10**, 718—757).—The theory that an increase in hardness of an alloy results from the highly dispersed precipitation of a soluble constituent from solid solution was first applied to explain the age-hardening of duralumin. The author gives a critical review of the precipitation theory and slip interference theory of hardness in the light of new experimental work. Dispersions of sufficient fineness are as a rule produced only by the decomposition of a supersaturated solid solution. In aluminium–copper alloys artificial ageing is due to the precipitation of very small particles of $CuAl_2$. In duralumin the particles are most probably Mg_2Si. Hardening during ageing increases as the quenching temperature increases, and coalescence of the fine particles causes a decrease of hardness. In Al–Mg–Si alloys the age-hardening effect was proportional to the amount of Mg_2Si dissolved during the solution heat treatment. New types of high-strength aluminium alloys are described. The hardening of magnesium–aluminium alloys is described, the maximum hardening occurring at 12% Al. Copper–iron alloys showed no appreciable age-hardening. In $\alpha\beta$-brass (56·6% Cu) hardening results were obtained from the precipitation of a constituent softer than the matrix in which it is precipitated. Age hardening was found in lead–antimony alloys containing over 0·5% Sb. Precipitation hardness was found to occur in iron–tungsten alloys which retained red-hardness and could be used as high-speed tools. T. H. BURNHAM.

Thermo-electric power of metallic aggregates. G. MASING (Wiss. Veröff. Siemens-Konz., 1926, **5**, 175—182).—A mathematical paper in which it is shown that the thermo-electric power of aggregates of two metals must lie between the values for each individual metal, and is directly proportional, or very nearly so, to the composition of the aggregate, the actual value depending on the electrical conductivity and heat conductivity of the components. A. R. POWELL.

Silumin and its structure. B. OTANI (Sci. Rep. Tohoku, 1926, **15**, 679—719).—See B., 1926, 831.

PATENTS.

Preventing the dissolution of iron and steel in

sulphuric acid and pickling baths. J. G. SCHMIDT and H. R. LEE, Assrs. to THE NEWPORT CO. (U.S.P. 1,608,622, 30.11.26. Appl., 30.9.25).—Iron and steel products may be pickled in a bath containing sulphuric acid and a thiourea compound. M. E. NOTTAGE.

Manufacture of metal alloys. SIEMENS & HALSKE A.-G., Assess. of E. DUHME (U.S.P. 1,607,245, 16.11.26. Appl., 7.7.25. Conv., 2.8.24).—The constituent metals are dissolved electrolytically or otherwise in mercury, the mercury is removed by heating the amalgam under reduced pressure, and the porous product fused. The process is of particular value when one of the constituent metals has a high m.p. T. S. WHEELER.

Process and apparatus for treating minerals. M. STOCKTON, Assr. to CELITE CO. (U.S.P. 1,611,791, 21.12.26. Appl., 20.7.25).—In a milling and drying process, loss of dust is prevented by disintegrating the material in the presence of moisture. The disintegrated material is then aspirated into a separating apparatus from which the dust-free air and the disintegrated material are separately discharged, the latter into a heated gas. The mixture of gas and disintegrated material is then passed into a second separating apparatus from which the partly-spent gas containing dust is again circulated to the moist disintegrated material. M. E. NOTTAGE.

Metallurgical treatment of copper silicate ores or products. W. DEWAR (E.P. 262,552, 6.10.25).—The copper present is converted into oxide, soluble in an ammoniacal solution or solvent, by heating the crushed ore to a carefully regulated temperature in an atmosphere best suited to the particular class of material. *E.g.*, the ore may be heated at 190—230° in the presence of producer gas or at 500—550° in air or other neutral atmosphere. The duration of treatment and variation in temperature depend upon the size of the particles, the percentage of copper in the ore, and its varying state of chemical combination. The material is then leached by ammoniacal solutions or solvents, the copper and ammonium salts being recovered from the solution.
M. E. NOTTAGE.

Purification of waste gases [from lead-fume furnaces]. F. KRUPP GRUSONWERK A.-G. (E.P. 256,643, 6.8.26. Conv., 7.8.25).—The waste gases produced in a furnace used for the volatilisation of metals or metal compounds are freed from carbon and other impurities before being admitted into the purifying installation, by burning them in the furnace flue in which an auxiliary heating device is provided, an additional purification of the volatile metal oxides being thus effected. The impurities deposited in the furnace flue are continuously returned to the furnace charging device.
M. E. NOTTAGE.

[Aluminium] alloy. F. P. DUSTAN, Assr. to J. WALRATH (U.S.P. 1,606,616, 9.11.26. Appl., 7.10.24. Renewed 14.9.26).—An alloy containing 84% Al, 10% Sn, and 6% Zn is light, durable, and easily machined.
T. S. WHEELER.

Light-metal alloy. H. H. DOW and J. A. GANN, Assrs. to THE DOW CHEMICAL CO. (U.S.P. 1,606,752, 16.11.26. Appl., 12.12.21).—An alloy of magnesium and zinc in solid solution, in which less than 0·5% Zn is present. H. ROYAL-DAWSON.

Recovery of zinc as sulphate from dross. Recovering metal [zinc]. J. O. BETTERTON, Assr. to AMERICAN SMELTING & REFINING CO. (U.S.P. 1,605,640—1, 2.11.26. Appl. [A, B], 28.6.23. Renewed, [A] 20.4.26; [B] 26.3.26).—(A) Dross from zinc-melting retorts, containing chiefly zinc, zinc oxide, zinc chloride, and ammonium chloride, is screened to remove oversize particles, which are returned to the retort, and is then calcined in contact with air so as to volatilise the metallic chlorides, and oxidise the metallic zinc and carbon present. The chlorides are condensed, and returned to the retort for use as a flux, and the zinc oxide is dissolved in dilute sulphuric acid. The solution obtained is sufficiently pure to be electrolysed. (B) The blue powder containing zinc, which is obtained in refining lead, is screened to remove oversize particles, which are returned to the main process, and is then heated in a retort with zinc chloride, which dissolves any zinc oxide in the mixture, and forms a slag, which is skimmed off the molten zinc, and calcined to give impure zinc oxide and volatilised zinc chloride. T. S. WHEELER.

Treatment of zinc waste. J. N. POMEROY (U.S.P. 1,609,882, 7.12.26. Appl., 6.5.22).—The chloride content of zinc waste may be lowered by mechanically separating the metallic zinc, treating the residue with sulphuric acid to remove the greater portion of the chloride, and then roasting it to volatilise more of the chloride.
M. E. NOTTAGE.

Iron alloys. L. CAMMEN (U.S.P. 1,612,570, 28.12.26. Appl., 26.3.26).—Iron alloys containing less than 0·03% C may be manufactured in the following stages: (1) the Bessemer stage, in which the blowing is continued until and beyond the end of the period of carbon burning; and (2) the furnace stage, in which the metal is held under such conditions that it cannot be contaminated by carbon, a temperature of 1550° or more being maintained.
M. E. NOTTAGE.

Manufacture of ferrotungsten. C. R. SCHROEDER, Assr. to METAL AND THERMIT CORP. (U.S.P. 1,609,969, 7.12.26. Appl., 31.10.23).—Silico-thermic reduction of iron–tungsten compounds is effected in the presence of an excess of an alkali halide flux capable of producing a highly fluid slag without the addition of an oxidising agent. M. E. NOTTAGE.

Condensing metallic vapours. NEW JERSEY ZINC CO., Assees. of G. T. MAHLER, E. C. HANDWERK, and E. H. BUNCE (E.P. 251,985, 1.5.26. Conv., 5.5.25).—The condensing chamber consists of a cylinder with vertical walls mounted on top of the furnace and in line with the retort, and communicating with the latter through a constricted nozzle or nozzles fixed in an annular plate which partially closes the bottom; each nozzle extends upwards about midway into the chamber, and the space between it and the cylinder forms a well in which the molten zinc collects, and from which it can be withdrawn through a tap-hole. The flat circular ceiling has an opening loosely covered by a plate. The chamber is surrounded by a layer of heat-insulating material, and by varying the thickness of this layer the temperature of the walls can be carefully controlled. The zinc or other metallic vapour mixed with carbon monoxide passes upwards from the retort through the nozzle, and

into the condensing chamber, where it impinges on the ceiling plate. The metallic vapour condenses as a film of molten zinc which flows down the walls and is collected in the well, the exhaust gases escaping through the loose plate at the top. M. E. Nottage.

Zinc alloys particularly suitable for casting. New Jersey Zinc Co., Assees. of W. McG. Peirce and E. A. Anderson (E.P. 252,020, 10.5.26. Conv., 11.5.25).—Magnesium not exceeding 0·3% in quantity is added to zinc alloys for use in casting or die casting; *e.g.*, such alloys may contain 0·01—0·30% Mg, and up to 15% Al, which may include copper. C. A. King.

Recovering float-gold and the like in suspension. J. E. Parker (U.S.P. 1,607,845. 23.11.26. Appl., 18.12.22).—Float-gold and other comminuted mineral values may be recovered from a stream of natural-flowing water by placing a viscous hydrocarbonaceous mass in the stream on immobile rigid supports of combustible material, maintaining these in contact with the flowing water until a substantial quantity of the comminuted values have coalesced with the mass, removing the latter from the stream, and finally burning the supports, thus leaving the mineral values intact. M. E. Nottage.

Process for recovering precious metals. P. Kuehn (U.S.P. 1,606,249, 9.11.26. Appl., 12.10.22).—Precious metals may be recovered from low-grade ores by agitating the finely divided material in the presence of soapy water with a current of air. After passing from the solution, the air and any matter carried with it is brought into contact with mercury, which removes the metallic content. M. E. Nottage.

Flotation apparatus [for ores]. J. C. MacIntosh, Assr. to General Engineering Co. (U.S.P. 1,608,896, 30.11.26. Appl., 13.11.25).—The apparatus consists of a cell adapted to retain a charge of pulp, and has a means of froth discharge, a rotor composed partly of finely porous material, means for turning the rotor about a horizontal axis, and for passing a gas into the rotor and out again through the porous material. M. E. Nottage.

Production of protective coatings [on metals]. C. G. Fink and L. C. Pan, Assrs. to Chemical Treatment Co. (U.S.P. 1,606,159, 9.11.26. Appl., 18.12.25).—A foundation metal plated with a protective metal is immersed in a bath of molten lead, in order to eliminate "pin-hole" defects in the plate without producing discoloration of the protective metal. J. S. G. Thomas.

Furnace for melting metals. J. C. Greenway (U.S.P. 1,608,557, 30.11.26. Appl., 22.7.25).—The furnace consists of an oversize hearth and a roof having pendant side walls, the lower ends of which overlie the hearth in spaced relation to it and terminate above the slag line of the furnace. The lower ends of the side walls and the hearth define between them charging spaces, opening into the furnace below the side walls. Means are provided for raising and lowering the side walls and roof and supporting them in any vertical adjustment. M. E. Nottage.

Metal melting furnaces. L. F. Tooth (E.P. 261,819, 29.7.25).—In a furnace in which metal, *e.g.*, lead, is melted, and run from a pot provided with a valved outlet, a removable spout conveys the metal from a guiding shoe under the valve through the wall of the furnace. The opening in the furnace wall is lined with a tubular casting of U section, the end of which is closed during the intervals between the periodic discharges of molten metal to avoid oxidation of the metal near the outlet. C. A. King.

Reverberatory furnaces [for metals]. F. Wüst (E.P. 243,711, 17.11.25. Conv., 27.11.24).—A furnace for melting metals or alloys, in which the raw material is preheated and melted by gases passing out through the charging shoot, has a cavity provided in the rear wall of the charging shoot where the floor of the gas outlet passage meets it. Thus the heating gases from the hearth can penetrate into this portion, and the cooling and solidification of material at this point is avoided. M. E. Nottage.

Bright-annealing furnaces. Siemens-Elektrowarme-Ges.m.b.H. (E.P. 252,405, 25.5.26. Conv., 25.5.25).—The furnace consists of separate upper and lower parts; the material to be annealed is placed on the lower part and surmounted with a bell filled with a protective gas, the lower edge of the bell being closed with a sand seal. The upper part can be turned down over the protective bell prior to the annealing operation, and lifted off at the end of it; or the upper part can be stationary and the lower part removed. Means are provided for removing the protective gas by suction and conducting it to a second furnace or to a gas-holder. Upon termination of the annealing process, the heated upper part is replaced by a cold upper part and the former placed over another protective bell, which is to be heated, whilst a current of air is circulated between the upper part and the protective bell in order to accelerate the cooling process. M. E. Nottage.

Electric resistance furnace for bright annealing. Siemens-Schuckertwerke Ges.m.b.H., Assees. of Heraeus-Vacuumschmelze A.-G. (E.P.244,425, 28.10.25. Conv., 15.12.24).—In an electric resistance furnace for bright annealing the furnace casing is closed permanently at one end and, by means of several separate casings, made gas-tight. An inert atmosphere is maintained in the furnace, and means are provided for transferring the charge from the heated to a cooling zone. C. A. King.

Annealing and like furnaces. C. F. Kenworthy (E.P. 262,651, 14.5.26).—One heating chamber and several cooling chambers are built in a circle over another larger chamber. The goods in containers are placed on a rising, falling, and rotating platform contained in the large lower chamber. The containers visit each smaller chamber in succession, being discharged and re-charged immediately before being heated. The heating chamber may be constructed with walls in two layers—heat-resisting and heat-insulating—and to prevent steam or other air-excluding gas from affecting the heat-insulating lining a metallic lining may be inserted between the two. B. M. Venables.

Electrochemical processes and apparatus for the extraction of copper and zinc from ores. H. S. Mackay (E.P. 262,546, 24.9.25 and 4.11.25).—The copper

and zinc contained in the ores are obtained in solution as sulphates, and copper is continuously deposited electrolytically from the solution until the concentration of copper sulphate is low relatively to the zinc sulphate in the solution, which is then evaporated in order to crystallise out a portion of the zinc sulphate. The residual solution is further electrolysed and evaporated.
J. S. G. Thomas.

Electrolytic refining of metals. F. F. Colcord, Assr. to United States Smelting, Refining, & Mining Co. (U.S.P. 1,607,869—70, 23.11.26. Appl. [a], 1.6.25; [b], 2.1.26).—(a) Sulphite lignose is added to an acid electrolyte. (b) Water-soluble hydroxy-derivatives of benzene are added to the electrolyte containing sulphuric acid used in the electrodeposition of zinc.
J. S. G. Thomas.

Process and apparatus for electrolytic refining. F. F. Poland, Assr. to American Smelting and Refining Co. (U.S.P. 1,609,771, 7.12.26. Appl., 11.8.25).—Anodes and cathodes are alternately spaced at a distance less than the normal thickness of irregularities produced on the starting sheet during electrolysis, and insulating spacing devices of sufficient rigidity to prevent buckling of the starting sheet are arranged between the anodes and the starting sheet. These devices are removed when the cathode sheets have become sufficiently built up to render them rigid. J. S. G. Thomas.

Method and means for electrodepositing nickel metals and the resulting products. Means for electrodepositing metals. C. P. Madsen, Assr. to Madsenell Corp. (U.S.P. 1,607,960 and 1,607,994, 23.11.26. Appl., [a] 26.6.23, [b] 4.8.21. Renewed 4.6.26).—(a) A "nickel metal" electrodeposition bath containing an iron compound or compounds is maintained substantially free from ferric iron compounds during the process of electrodeposition, so that the deposited metal is substantially free from pits. (b) A bath for electrodepositing "nickel metals" is treated with chlorine to oxidise any organic matter present. J. S. G. Thomas.

Treatment of iron or steel for preventing oxidation or rusting. T. W. Coslett (U.S.P. 1,610,362, 14.12.26. Appl., 6.2.26. Conv., 26.6.25).—See E.P. 247,071; B., 1926, 283.

Addition of aluminium to aluminium-containing alloys. W. A. Mudge, Assr. to Internat. Nickel Co. (U.S.P. 1,612,642, 28.12.26. Appl., 16.6.25).—See E.P. 253,879; B., 1926, 884.

Separation and recovery of arsenic and tin. Vulcan Detinning Co., Assee. of J. A. Lahey (E.P. 251,932, 9.10.25. Conv., 6.5.25).—See U.S.P. 1,575,217; B., 1926, 360.

Process of refining tin. L. Schertel and W. Lüty (U.S.P. 1,609,967, 7.12.26. Appl., 27.4.23. Conv., 29.4.22).—See E.P. 196,938; B., 1924, 339.

Enamelling of cast and sheet metal (E.P. 262,159).—See VIII.

[Apparatus for the] gravity concentration of ores. F. H. Cothay, Assr. to Ropp Tin, Ltd. (U.S.P. 1,611,339, 21.12.26. Appl., 18.8.24. Conv., 14.5.24).—See E.P. 238,297; B., 1925, 812.

Welding of iron or steel articles. Metals Protection Corp., Assees. of C. H. Humphries (E.P. 257,571, 29.3.26. Conv., 31.8.25).

XI.—ELECTROTECHNICS.

Patents.

Electric furnace. British Thomson-Houston Co., Ltd., Assees. of J. A. Seede (E.P. 249,566, 23.3.26. Conv., 23.3.25).—An electric arc furnace in which a predetermined length of arc is automatically maintained is provided with an inner chamber for the electrodes, and with means whereby the point of maximum temperature may be adjusted relatively to the charge.
J. S. G. Thomas.

[Varying the resistance of] electric furnaces. Studien-Ges. für Wirtschaft u. Ind. m.b.H. (E.P. 254,756, 27.8.25. Conv., 27.2.25. Addn. to 239,222; B., 1925, 927).—In a furnace constructed in accordance with the previous patent, the resistance is contained in an open refractory channel having holes or tapping points through which resistance material may be introduced or withdrawn in order to increase or reduce the resistance. J. S. G. Thomas.

Electric incandescence lamp. H. Wade. From N. V. Philips' Gloeilampenfabr. (E.P. 262,288—9, 20.1.26).—(a) An electric incandescence gas-filled lamp is filled with nitrogen, *e.g.*, at a pressure of 50—60 cm. of mercury, and a small quantity of halogen vapour, *e.g.*, iodine. (b) Evaporation of the tungsten filament of an electric incandescence lamp is reduced by using a salt of hydrofluoboric acid, *e.g.*, potassium boron fluoride, as a "getter" in the lamp. J. S. G. Thomas.

Incandescence lamp [filament]. O. V. Maurer (U.S.P. 1,612,459, 28.12.26. Appl., 17.7.25).—A filament for electric lamps consists of an alloy containing 50—99% Ta and 1—50% W. F. G. Crosse.

Introduction of hygroscopic material into evacuated devices [electric lamps]. W. C. Sproesser, Assr. to Westinghouse Lamp Co. (U.S.P. 1,612,661, 28.12.26. Appl., 16.7.23).—The filament of an electric lamp is "gettered" with a non-hygroscopic product of phosphorus pentoxide which is decomposed when heated to incandescence. F. G. Crosse.

Manufacture of vacuum-tube filaments. F. S. McCullough (U.S.P. 1,610,560, 14.12.26. Appl., 24.10.22).—A portion of a coating deposited upon a metal base is reduced to the metallic state and fused to the base, so that craters extending into the surface of the base are formed. J. S. G. Thomas.

Electric [arc] lamp. P. K. Devers, jun., Assr. to General Electric Co. (U.S.P. 1,610,077, 7.12.26. Appl., 1.6.23).—Mercury vapour is supplied to an arc struck between electrodes, and a neutral electrode of substantially the size of these electrodes and capable of selective radiation near the red end of the spectrum is arranged in the path of the arc. J. S. G. Thomas.

Electric battery. H. T. Harrison and G. Campbell (E.P. 262,016, 13.1.26).—In an electric battery comprising a number of cells in a normally closed container, the walls separating the cells do not extend to the top of

the container, but leave a space above and common to all the cells, to facilitate simultaneous filling to the same level. A sludge-collecting trough provided with a filter is arranged in the space above the cells.
J. S. G. Thomas.

Electrolytic plating machine. J. Hulmer (U.S.P. 1,609,357, 7.12.26. Appl., 19.11.25).—A rotatable shaft is arranged on a solution tank and carries a tumbling barrel turning through the solution within the tank. A cathode is arranged on the inner periphery of the barrel, and anodes comprising separable clamping wheels are carried by the shaft. J. S. G. Thomas.

Paper insulation for electric cables etc. Felten u. Guilleaume Carlswerk A.-G. (E.P. 251,931, 30.9.25. Conv., 8.5.25).—Graduation in the dielectric constants of paper layers for insulating cables is obtained by compressing the layers to different extents, impregnating them with substances of different dielectric constants, such as artificial or phenol resins, or by a combination of both methods. B. P. Ridge.

Primary and secondary electric cell. A. Cellino (U.S.P. 1,609,692, 7.12.26. Appl., 6.11.24. Conv., 22.11.23).—See E.P. 225,197; B., 1925, 458.

Electric furnaces, ovens, and other heating chambers. N. E. North (E.P. 262,859).

[Plates for] secondary batteries. J. Stone & Co., Ltd., and A. H. Darker (E.P. 262,528, 14.9.25).

Manufacture of vitreous containers for electric lamps, thermionic valves, etc. A. Emerson, jun. (E.P. 262,498, 3.9.25).

Method of enhancing the therapeutic value of ultra-violet rays. Hanovia Chemical and Manuf. Co. (E.P. 262,703, 16.10.26. Conv., 1.5.26).

Glass for insulating purposes (E.P. 256,189).—See VIII.

Recovery of ingredients of waste lead battery paste (U.S.P. 1,610,899).—See VII.

Method for counteracting scaling and corrosion (U.S.P. 1,611,853).—See X.

XII.—FATS; OILS; WAXES.

Properties of "mickerfett" [the mesenteric fat of the pig]. H. Jesser (Z. angew. Chem., 1926, **39**, 1551—1552).—In cases of suspected adulteration of lard with beef or mutton tallow it has been stated that the abnormal chemical and physical properties observed were rather due to the presence of "mickerfett" or mesenteric pork fat. A number of pigs were therefore slaughtered, and the fat from different parts was tested separately for refraction, acidity, and the Polenske and Bömer difference numbers. The results are tabulated. The mickerfett is markedly harder than fat from the back or belly, and is distinguished by higher acidity. The Polenske and Bömer difference numbers are, however, in agreement with those of other kinds of pork fat, and no confusion with tallow is likely. C. Irwin.

Application of ultra-violet light [in analysis of fats etc.] A. van Raalte (Chem. Weekblad, 1926, **23**, 580—581).—Lard shows no luminescence in ultra-violet radiations, whereas refined pig's fat exhibits a violet luminescence. Plant cell-wall material also luminesces strongly; the use of the quartz lamp in microscopical examination of plant products would therefore be valuable. S. I. Levy.

Comparison of deflocculating powers of soaps by the carbon-black test. R. M. Chapin (Ind. Eng. Chem., 1926, **18**, 1313—1316).—A modification of the McBain, Harborne, and King carbon-black test (J.S.C.I., 1923, **42**, 373 T) has been employed to examine the effects of various factors on the deflocculating powers of soap solutions. Soap filtrates in all the experiments are referred back colorimetrically to standard filtrates from a strictly neutral solution of pure potassium palmitate at 40°, and the value of each is given as the "potassium palmitate ratio." A series of experiments, the results of which are expressed in graphs, demonstrates that at equilibrium, colloidal soap is inert, simple soap anions are the deflocculating agent at the lower concentrations of total soap, simple soap molecules may be the agent at the higher concentrations (these two being sharply distinct), and that the transformation of colloidal soap into soap crystals gives an increase in deflocculating power. These conclusions are deduced in part from the indubitable fact that the deflocculating power of soaps in these tests reaches distinct maxima at two different concentrations. Further work both with "naked" and "oiled" carbon black is promised. E. Holmes.

***iso*Propyl alcohol as a substitute for ethyl alcohol. III. Determination of acid numbers.** H. A. Schuette and M. P. Smith (Ind. Eng. Chem., 1926, **18**, 1242—1245).—A modification of the method of determining the acidities of fatty oils and waxes, using *iso*propyl alcohol as solvent, is proposed. A solution of 28·2 g. of the sample in 50 c.c. of the alcohol is titrated with a 0·1*N*-solution of potassium hydroxide in the same solvent, using phenolphthalein as indicator. The results obtained appear to compare favourably with the control experiments using the ethyl alcohol method when the test samples have acid numbers below 8. Above this the method gives erratic results except in the case of the linseed oil group. Advantages claimed for the method are: the complete dispersion of the fatty oil in the solvent, the excellent degree of miscibility of the products of the reaction in the solution of the test sample, and the sharpness of the end point. Satisfactory results are also obtained by dissolving the samples in other solvents, *e.g.*, light petroleum, ethyl ether, acetone, benzene, a mixture of benzene and *iso*propyl alcohol (1 : 1), and titrating with the above-mentioned solution of alkali. Evidence is adduced to show that partial saponification of the fatty oil or wax during neutralisation is a contributory cause of some of the high results. E. Holmes.

Standard methods for the sampling and analysis of commercial fats and oils. American Chemical Society Committee on Analysis of Commercial Fats and Oils (Ind. Eng. Chem., 1926, **18**, 1346—1355).—This revision of the A.C.S. Committee's first report of 1919 gives the precise detailed methods for the sampling and examination of commercial fats and oils as unanimously adopted in America in February, 1926. It is noteworthy that the Committee now considers the Wijs

method superior to that of Hanus for the determination of the iodine value of linseed and other oils, and quite as satisfactory as the Hübl method for tung oil. No methods have yet been developed with special reference to the waxes. E. HOLMES.

Polymerisation during the drying and boiling of fatty oils, with reference to the work of Auer. J. SCHEIBER (Chem. Umschau, 1927, **34**, 6—8).—Mainly polemical with Auer (B., 1926, 450, 888), regarding the validity of the chemical rather than the colloidal theory of the drying and thickening of fatty oils.
E. HOLMES.

Method of detecting and determining tung oil. I. J. N. GOLDSMITH (J. Oil Col. Chem. Assoc., 1926, 9, 342—351).—If a paint medium or varnish, thinned with light petroleum alone or mixed with a small percentage of absolute alcohol or methylated ether, be treated with excess of nitrous acid or nitrous fumes, any tung oil present is converted into a practically insoluble yellow solid. The presence of amyl acetate, ethyl acetate, chloroform, methyl alcohol, ethyl alcohol, benzene, or nitrobenzene may prevent the precipitation. Originally used as a qualitative test, the method provides a rough quantitative determination if the washed precipitate be dried to constant weight in coal gas or carbon dioxide at 100°, or under a reduced pressure of about 30 mm. at 40°. As precipitating agent, pure nitrogen peroxide (from lead nitrate) was found to be even more reactive than nitrous fumes, whereas pure nitric oxide produced no precipitate in the absence of air, but a dense white precipitate on exposure to air. An oil is extracted on washing a pure tung oil precipitate, the suggested source of which is the olein originally present; the remainder is thought to be β-elæostearin containing varying amounts of nitrogen oxides. The precipitate dried to constant weight is shown to be a decomposition product, and the numerical results are admittedly empirical. S. S. WOOLF.

Determination of the "break" (and foots) in linseed oil. G. S. JAMIESON and W. F. BAUGHMAN (J. Oil Fat Ind., 1926, 3, 307—309; Chem. Abstr., 1926, **20**, 3825).—10 g., transferred to a separating funnel with 50 c.c. of petroleum b.p. $< 80°$, are shaken (3 min.) with 10 c.c. of 14% potassium hydroxide solution and then with 25 c.c. of 50% ethyl alcohol (20 sec.). The lower layer and precipitate, after extraction three times with 20 c.c. of petroleum, are used for the determination of the fatty acids. The petroleum extracts and washings, after washing with alcohol, are evaporated, the residue of neutral oil being heated to constant weight in an atmosphere of carbon dioxide at 120—125°. The alkali solution, after removal of the alcohol at 100°, is diluted with 75 c.c. of water and acidified with hydrochloric acid, and the cooled liquid filtered; the acids are then extracted with petroleum as before. The percentage of "break" is obtained by subtracting the percentages of neutral oil and fatty acids from 100.
A. A. ELDRIDGE.

***iso*Propyl alcohol as a substitute for ethyl alcohol. I. Determination of saponification values.** H. A. SCHUETTE and L. E. HARRIS (J. Amer. Pharm. Assoc., 1926, **15**, 166—173; Chem. Abstr., 1926, **20**, 3827).—*iso*Propyl alcohol is a convenient substitute for ethyl alcohol. It is preferred for its freedom from aldehydes and for the rapidity of saponification.
A. A. ELDRIDGE.

Detection of adulteration of essential oils [and fatty oils] with alcohol. DÁVID.—See XX.

Solubility of alkaloids in oils. TELLERA.—See XX.

PATENTS.

Method and apparatus for melting blocks of hard fats. B. JIROTKA (E.P. 262,643, 17.4.26).—In order to break up and melt hard fat without forcing it through disintegrating mechanism, the blocks of fat are placed on a grid and reduced in size by heat and/or mechanical means to pieces that will fall through the bars of the grid into a melting chamber below.
S. S. WOOLF.

Manufacture of polish and waterproofing composition. C. H. THOMPSON and W. J. MCGIVERN (E.P. 262,371, 6.8.25).—The molten mixture of a mineral wax with a fatty acid or resin is emulsified with an aqueous solution of an organic colloid and an alkali.
S. S. WOOLF.

Purification of wool fat. S. FOWLER and E. EDSER (U.S.P. 1,610,854, 14.12.26. Appl., 13.8.26. Conv., 24.3.25).—See E.P. 253,995; B., 1926, 759.

Manufacture of fatty acids. A. WELTER (U.S.P. 1,612,682, 28.12.26. Appl., 4.10.24. Conv., 23.10.23).—See E.P. 223,898; B., 1925, 557.

Manufacture of artificial leather (E.P. 261,863).—See XV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Separation and analysis of pigments in lacquer. F. W. HOPKINS (Ind. Eng. Chem., 1926, **18**, 1233).—A favoured method of isolating the pigment portion of a lacquer is to precipitate pigments, nitrocellulose, plasticisers, and resins by the addition of methylene chloride. After removal of the solvents by decantation or by evaporation, the nitrocellulose, plasticisers, and resins are extracted by a mixture of equal parts of benzene and ethyl acetate, and the separated pigment is dried. General details of the determination of pigments usually found in lacquers are briefly indicated.
S. S. WOOLF.

Standardisation and measurement of colour. F. A. O. KRÜGER (Farben-Ztg., 1926, **32**, 682—685).—The general account of colour standardisation by the methods of Ostwald and the German Laboratory for Colour Science (as issued by the latter in 1925) is reprinted. The drawbacks of the "colour circles" produced by Ostwald, particularly the variation in the standard colours, are avoided in the use of a new "tint-photometer" (devised by C. Pulfrich), in conjunction with permanent colour strips prepared by the above laboratory. Difficulties due to the nature of the coloured surface to be examined are obviated, and increased simplicity and accuracy are claimed. Uses of the tint-photometer other than the simple measurement of colour, *e.g.*, the grading of white pigments, the measurement of opacity, etc., are described.
S. S. WOOLF.

Separation and analysis of the volatile solvents and thinners of lacquers. R. M. CARTER (Ind. Eng. Chem., 1926, **18**, 1234—1235).—The author recommends the following method:—About 200 c.c. of lacquer are distilled from a flask immersed nearly to the side arm in an oil bath maintained at 120° (higher temperatures leading to possible decomposition of nitrocellulose and consequent vitiation of results). When the distillate ceases to come over at 120°, steam is used to bring over the higher-boiling solvents and other materials volatile in steam, which are then separated from the water layer by saturation of the latter with sodium sulphate or calcium chloride. The fractions are examined for odour, sp. gr., boiling range, saponif. value, and solubility in concentrated sulphuric acid and in water.
S. S. WOOLF.

Synthetic resins containing nitrogen. A. EIBNER and E. KOCH (Z. angew. Chem., 1926, **39**, 1514—1518).—As well as the artificial resins of coumarone and phenol large numbers of resins from primary aromatic amines and aliphatic aldehydes have been prepared. The "resinophoric" groups which enable these compounds to polymerise are discussed, and it is shown that the azomethene grouping in Schiff's base is not always "resinophoric." Schultz base, made by treating aniline hydrochloride with acetaldehyde, forms an amorphous substance of resinous character, and the authors have prepared two such compounds, m.p. 175—185° and 250°; mol. wt. determinations are given, and investigations of the nitroso-derivative. The resin obtained from Schultz base is suitable for paints and varnishes.
W. G. CAREY.

Acid number of Peru balsam. N. SCHOORL and J. KUIJLMAN (Pharm. Weekblad, 1926, **63**, 1425—1428).—The Dutch pharmacopœia employs for the determination addition of excess of 0·1*N*-ammonia solution, and back titration with acid. It is found that borax is as suitable as ammonia for this purpose, and its use is advocated in view of the greater stability of the standard solutions.
S. I. LEVY.

Method of detecting and determining tung oil. GOLDSMITH.—See XII.

Determination of the "break" (and foots) in linseed oil. JAMIESON and BAUGHMAN.—See XII.

PATENTS.

Manufacture of luminous paint. E. G. COLTON (U.S.P. 1,610,747, 14.12.26. Appl., 16.11.21).—A mixture including a large proportion of a precipitated zinc sulphide, a small proportion of sodium chloride, and a relatively small amount of a mixture of mesothorium and radio-thorium is claimed.
S. S. WOOLF.

Condensation products from mono- and di-cyclic phenols and aldehydes. CHEM. FABR. DR. K. ALBERT G.M.B.H., A. AMANN, and E. FONROBERT (E.P. 261,472, 19.8.25).—Definite phenol alcohols, free from resinous and other undesired by-products, are obtained by slowly adding a mono- or di-cyclic phenol (usually in alkaline solution) to a quantity of aldehyde at least equal to the theoretical maximum amount that can attach itself to the phenol. Such condensation products are particularly suitable for conversion into synthetic resins.
S. S. WOOLF.

Processes of making moulded articles. BRITISH THOMSON-HOUSTON CO., LTD., Assees. of C. F. PETERSON (E.P. 250,942, 13.4.26. Conv., 14.4.25).—The homogeneity, mechanical strength, and insulating properties of articles made from mica-flakes with a "glyptal" (glycerol-phthalic anhydride) binder or other similar moulding composition are improved by carrying out the first part of the "curing" of the resin under such conditions that the escape of the volatile by-products is facilitated. This is achieved by heating in a vacuum and/or by the application of intermittent pressure before the final polymerisation.
S. S. WOOLF.

Plasticising phenolic moulding materials. F. P. BROCK, Assr. to BAKELITE CORP. (U.S.P. 1,609,506, 7.12.26. Appl., 2.11.23).—The plasticity of phenol resin compositions of the potentially reactive type is improved by incorporation of furfuraldehyde before hot moulding.
S. S. WOOLF.

Manufacture of phenol resins. C. KULAS and C. PAULING, Assrs. to C. KULAS (U.S.P. 1,609,367, 7.12.26. Appl., 30.8.23. Conv., 12.10.22).—A methylene diphenol mixture, obtained by the action of acid on phenol and formaldehyde, is boiled with phenol alcohols, and the reaction product is dehydrated and heat "cured." (Cf. G.P. 431,619; B., 1926, 890.)
S. S. WOOLF.

Manufacture of linoleum cement. P. SLANSKY, Assr. to DEUTSCHE LINOLEUM-WERKE HANSA (U.S.P. 1,610,331, 14.12.26. Appl., 27.6.25. Conv., 6.2.24.)—See E.P. 248,974; B., 1926, 451.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Microscopic investigation of rubber. H. DANNENBERG (Kautschuk, 1926, 276—277).—Apparatus is described for the microscopic and ultra-microscopic examination of rubber samples maintained at a thinness of approx. 20 μ by pressure. Typical photomicrographs are reproduced.
D. F. TWISS.

Structure of rubber. J. BEHRE (Kautschuk, 1926, 278—281).—Various technical processes such as coagulation of latex, mastication, calendering, and vulcanisation are reviewed in the light of the modern views as to the structure of rubber.
D. F. TWISS.

Stretched rubber, its "fusion line," and its density. H. FEUCHTER (Kautschuk, 1926, 260—263, 282—286).—A study of the anisotropy of unvulcanised rubber stretched while warm, particularly with respect to its rate of recovery at higher temperatures ("fusion line") and its sp. gr. (cf. B., 1925, 515).
D. F. TWISS.

Resin oils as softening agents [for rubber]. F. HEBLER (Gummi-Ztg., 1927, **41**, 761—762).—For use as a softening agent for rubber, resin oil should have *d* 0·960—0·990 and Engler viscosity (at 93—95°) 1·5—2·3; it should not show an evaporation loss exceeding 1% in 3 hrs. at 93—95°, and its swelling power for rubber should be as high as possible. The customary analytical tests are of minor value.
D. F. TWISS.

PATENTS.

Process for the production of rubber. H. BECKMANN (E.P. 262,179, 2.9.25. Addn. to 240,430; B., 1925, 1000).—In the manufacture of porous rubber by

vulcanising a gelled mixture of latex and vulcanising agent, the homogeneous jelly can be produced by the action of a gaseous or vaporised acid, *e.g.*, sulphurous acid, on the mixed latex; alternatively, the mixed latex may first be made to thicken by the addition of alum or of a salt of zinc or iron, when formation of the jelly may be completed with a liquid acid, *e.g.*, aqueous sulphurous acid. D. F. TWISS.

Treating caoutchouc latex. A. BIDDLE, Assr. to UNITED PRODUCTS CORP. OF AMERICA (U.S.P. 1,607,585, 16.11.26. Appl., 4.12.24).—An alkaline or lime solution of casein is mixed with caoutchouc latex and then precipitated by the addition of an excess of an acid substance. D. WOODROFFE.

Treating rubber and similar materials. T. WHITTELSEY and C. E. BRADLEY, Assrs. to NAUGATUCK CHEMICAL Co. (U.S.P. 1,607,331, 16.11.26. Appl., 24.9.25. Cf. U.S.P. 1,559,393; B., 1926, 68).—Rubber is treated with the reaction product of ammonia and a sulphur halide and then vulcanised. D. WOODROFFE.

Production of vulcanised rubber and accelerators therefor. H. W. ELLEY, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,610,216, 7.12.26. Appl., 25.2.25).—Rubber is vulcanised with the aid of an accelerator containing the grouping

$$S:\dot{C}\cdot NR\cdot CR'R'''\cdot NR''\cdot \dot{C}:S$$

where R aud R'' represent hydrocarbon radicals with from 1 to 7 carbon atoms, R' and R''' represent hydrogen or hydrocarbon radicals with from 1 to 4 carbon atoms, and each of the two terminal carbon atoms (shown attached to sulphur) is attached to another sulphur atom. D. F. TWISS.

Manufacture of dry well-preserved rubber from latex. E. S. A. COHEN (U.S.P. 1,610,226, 14.12.26. Appl., 31.3.24. Conv., 12.4.23).—See E.P. 214,210; B., 1925, 368.

Mixing substances with rubber latex. E. HOPKINSON, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,611,349, 21.12.26. Appl., 9.2.22).—See E.P. 193,044; B., 1924, 480.

Treatment of zinc oxide [for rubber compounding] (E.P. 253,875).—See VII.

Production of leather compounds [containing rubber] (E.P. 261,879).—See XV.

Concretes, cements, etc. (U.S.P. 1,610,864).—See IX.

XV.—LEATHER; GLUE.

PATENTS.

Production of leather compounds. R. MEYER (E.P. 261,879, 28.9.25).—Air-dried leather is dried at about 50° and compounded with rubber, with or without a vulcanising agent, precautions being taken to prevent ingress of moisture between the drying and compounding. 10 pts. of leather or leather waste may be reduced to fibre, dried, and incorporated with 1 pt. of raw rubber in 10 pts. of benzene to form a paste which is kneaded and moulded. D. WOODROFFE.

Manufacture of artificial leather and the like. G. E. SCHARFF, and NOBEL'S EXPLOSIVES CO., LTD. (E.P. 261,863, 3.9.25).—A small quantity (2%) of aromatic nitro-compound containing no hydroxyl group is mixed with the vegetable oils used as ingredients of plastic compositions for coating fabric, paper, and the like to prevent rancidity. D. WOODROFFE.

Evaporating apparatus [for tannin extracts] (E.P. 262,608).—See I.

XVI.—AGRICULTURE.

Determination of easily-soluble phosphoric acid in soil. H. NEUBAUER (Mitt. deut. Landw. Ges., 1926, 368; Bied. Zentr., 1926, **55**, 533—535).—A reply to Gerlach and Nolte (see B., 1927, 87).—The author holds that the "seedling" method for determination of readily available phosphoric acid in soil, although it may be further developed, has passed the experimental stage and can be applied directly in practice. It gives a measure of the stock of immediately available plant nutrients in the soil, and the results are therefore not directly comparable with field and pot experiments which aim at determining an undefined "fertiliser requirement" of soils. The pot experiments of Gerlach and Nolte supply evidence in favour of the usefulness of the "seedling" method. C. T. GIMINGHAM.

Determination of phosphoric acid content or phosphoric acid requirement of soils by the methods of Neubauer and of Lemmermann. O. ENGELS (Z. Pflanz. Düng., 1926, **B5**, 537—546; cf. B., 1926, 600).—Further experiments with a variety of soils show, with some irregularities, a general agreement between the results obtained by the Neubauer "seedling" method and by Lemmermann's "relative solubility" method (determination of the ratio of the P_2O_5 soluble in 1% citric acid to that soluble in hot 10% hydrochloric acid). Lemmermann's method is considered an important advance in the chemical examination of soils. C. T. GIMINGHAM.

Assimilation of phosphorus from phytin by oats. A. L. WHITING and A. F. HECK (Soil Sci., 1926, **22**, 477—493).—Experiments with oats in sand cultures showed that phosphorus in the form of phytin was assimilated more readily than in the form of inorganic phosphate, though, when given in large amounts, phytin proved harmful and reduced the yields. There was an increase in the phosphorus content of both grain and straw with increasing applications of phosphorus, whether in an organic or inorganic form. C. T. GIMINGHAM.

Absorption and utilisation of phosphoric acid and potassium by seedling plants. S. GERICKE (Z. Pflanz. Düng., 1926, **B5**, 550—553).—Preliminary experiments with rye seedlings grown in sand for 18 days show that, when phosphoric acid is given alone, 15·2% only is taken up by the plants, whereas, in the presence of calcium carbonate, the amount is increased to 39·3%. Corresponding figures for potassium absorption are 37·6% and 73·1%. If both nutrients are supplied, the percentage of K_2O taken up is slightly decreased, but that of P_2O_5 is not significantly changed. The influence of calcium carbonate is probably due to a change of reaction

favourable to the plants. The results are discussed with reference to Neubauer's method for determining readily available plant nutrients in soils. C. T. Gimingham.

Biochemical utilisation of the various forms of phosphoric acid in fertilisers. A. Strobel and K. Scharrer (Z. angew. Chem., 1926, 39, 1579—1584).—The action of many pure preparations of alkali and alkaline-earth phosphates, and of four commercial phosphatic fertilisers in promoting the growth of nitrifying bacteria has been examined; although no strictly comparable conclusions could be drawn as to the values of these compounds as fertilisers, it was established that their physiological action depended on their solubility, water-soluble phosphates having the most rapid action, followed by phosphates soluble in ammonium citrate solution, then by those soluble in citric acid. Tests on certain vegetables and cereals showed that the citrate-soluble phosphate of Rhenania phosphate gave as good a yield as the water-soluble phosphate of superphosphate fertilisers, whereas basic slag gave an appreciably poorer yield. Rhenania phosphate and superphosphate caused a much more rapid growth of root and stem in the young plant than basic slag; the efficiency of Rhenania phosphate, however, depends on its content of citrate-soluble phosphate. Tests on four different varieties of soils indicated that basic slag and dicalcium phosphate were always inferior to superphosphate and Rhenania phosphate. A. R. Powell.

Carbon dioxide production and gas permeability of soil. H. Lundegårdh (Arkiv Bot., 1923, 18, [13], 1—36).—Methods are described for the determination of the carbon dioxide emission from free soil surfaces, and of the absolute carbon dioxide production of soil at different depths. The free soil respiration, the absolute carbon dioxide production of the soil mass, the carbon dioxide content of soil air, and the permeability (soil respiration/soil air concentration) are mathematically related in a simple manner. Permeability is directly proportional to the diffusion coefficient; it is increased by cultivation and dryness and decreased by decreasing porosity and by dampness. The absolute carbon dioxide production nearly always increases with depth; it is accelerated by moisture so long as the permeability is not decreased, by deep intermixing of stable manure, by nutrient salts, and by improved ventilation. Hydrogen-ion concentration and humus content are not nearly related to the soil activity. The free soil respiration of unfertilised cultivated soils is about 0·4 g./m.^{2}/hr. A. A. Eldridge.

Preparation and effectiveness of basic copper sulphates for fungicidal purposes. E. B. Holland, C. O. Dunbar, and G. M. Gilligan (J. Agric. Res., 1926, 33, 741—751).—A number of basic sulphates, prepared by the action of alkaline-earth hydroxides and carbonates on copper sulphate, were investigated, with the object of producing a powder of good physical properties which could be used as a fungicide in the form of either spray-fluid or dust. A satisfactory product was obtained by stirring precipitated calcium carbonate with a solution of copper sulphate at 80°, filtering, and drying the precipitate at a low temperature. Both laboratory and field experiments with this material indicated that it was an effective fungicide, and easily applied as spray and dust. It is less adhesive and, in laboratory tests, somewhat less effective against the more resistant fungi than fresh Bordeaux mixture. C. T. Gimingham.

Water and water solutions of organic compounds as dips for the soil of potted plants infested with the Japanese beetle. W. E. Fleming (J. Agric. Res., 1926, 33, 821—828).—Larvæ of the Japanese beetle in the soil of small potted plants resisted asphyxiation in water for 15 days. No correlation was found between the rate of mortality of submerged larvæ and the concentration of dissolved oxygen in the water. Immersion of the soil in emulsions or solutions of various compounds was tested; carbon disulphide was the most effective, but insecticidal concentrations usually injured the plants. C. T. Gimingham.

Nutritive value of protein in beef extract, ox blood, ox palates, calf lungs, hog snouts, and cracklings. R. Hoagland and G. G. Snider (J. Agric. Res., 1926, 33, 829—843).—Feeding experiments with rats in which various meat products were the sole source of nitrogen in an otherwise adequate diet are reported. Rations containing 18% of "crude protein" (about 9% of true protein) in the form of beef extract, or 15% of protein as hog snouts, ox palates, or pork cracklings, were all inadequate for maintenance. The last three products consist mainly of connective tissue. The protein of calf lungs was of better quality, 10% being sufficient for moderate growth. Dried blood and hæmoglobin were very distasteful to rats. C. T. Gimingham.

Application of ultra-violet light [in plant analysis]. Van Raalte.—See XII.

Patents.

Method of promoting the growth of plants with carbon dioxide. E. Reinau (U.S.P. 1,611,072, 14.12.26. Appl., 6.2.23. Conv., 21.6.21).—The atmosphere in the neighbourhood of growing plants is enriched in carbon dioxide by burning readily ignitable carbonaceous matter which is made up into unit quantities so that any pre-determined quantity of carbon dioxide may be conveniently furnished. G. W. Robinson.

Method and apparatus for separating seed and other bodies. H. R. Warren, Assr. to Warren Seed Cleaning Co. (U.S.P. 1,609,636, 7.12.26. Appl., 7.4.22).—Materials of different sp. gr. are separated in an appropriate receptacle by the use of liquids of such densities that the materials to be separated arrange themselves at different levels. G. W. Robinson.

Preparation of an insecticide. W. Schmitz, Assr. to Tinolan Co. of America (U.S.P. 1,610,167, 7.12.26. Appl., 18.8.23).—Quillaia bark and seeds of the genus *Lupinus* are extracted with an inorganic acid. Sodium sulphate is added to the extract to form a solid mass, which is then pulverised. The product thus obtained consists of a sodium salt of an inorganic acid (including sodium sulphate) together with saponins from quillaia and salts of the alkaloids from the *Lupinus* seeds. G. W. Robinson.

Fumigating process [for grain]. E. C. Moffett, Assr. to American Cyanamid Co. (U.S.P. 1,613,186, 4.1.27. Appl., 7.5.24).—A portion of the grain is treated

with hydrocyanic acid, and distributed throughout the remainder of the grain by means of a current of air.

F. G. CROSSE.

Manufacture of fertilisers. E. L. PEASE and D. TYRER (U.S.P. 1,610,109, 7.12.26. Appl., 4.2.26. Conv., 19.2.25).—See E.P. 253,572; B., 1926, 717.

XVII.—SUGARS; STARCHES; GUMS.

Formation of sucrose in potatoes during drying. C. J. DE WOLFF (Chem. Weekblad, 1927, **24**, 18—19).—Tests with different varieties showed that sucrose is formed in all cases during drying if the potato is not too finely divided, the maximum formation being at about 16°; the reaction ceases when the content of dry material exceeds 53%. The quantity of sucrose obtained does not exceed 3% of the original weight of material, so that there is no present prospect of industrial utilisation of the process for sugar extraction.

S. I. LEVY.

Determination of lactose in bread. SNETHLAGE.—See XIX.

PATENTS.

Clarifying sugar solutions. K. SEO (U.S.P. 1,609,133, 30.11.26. Appl., 28.11.23. Conv., 30.11.22).—The sugar solution (molasses, cane or beet juice, etc.) is acidified or treated with water-soluble metallic salts, excepting those of the alkalis. A non-miscible fat solvent, *e.g.*, light petroleum, is then added and the whole agitated. On settling, the impurities rise to the surface in a gel form or are partly dissolved in the fat solvent, and are then removed by decantation or filtration.

F. R. ENNOS.

Production of dextrose from starch-bearing materials. CORN PRODUCTS REFINING CO., Assees. of W. B. NEWKIRK (E.P. 254,729, 11.8.25. Conv., 17.1.25. Cf. 232,938; B., 1925, 967).—Pure anhydrous dextrose is prepared from starch-converted dextrose containing less than 5% of impurities by dissolving it in water, bringing the solution to supersaturation, and crystallising at an initial temperature above 50°, without introduction of seed crystals. During crystallisation, the whole is agitated to ensure formation of granular crystals, since these may be completely freed from the mother liquor by centrifuging the resultant magma while in a semi-solid state.

F. R. ENNOS.

Manufacture of sugar. A. P. LEONARD (Re-issue 16,511, 28.12.26, of U.S.P. 1,568,512, 5.1.26).—See B., 1926, 294.

XVIII.—FERMENTATION INDUSTRIES.

Determination of lactose in bread. SNETHLAGE.—See XIX.

PATENTS.

Vinegar manufacture. F. NOLDIN, Assr. to P. HASSACK (U.S.P. 1,610,465, 14.12.26. Appl., 17.7.25. Conv., 6.5.25).—The liquid to be treated is placed in a closed vessel and forced by an oxidising gas into a porous medium, driving out the exhausted air. The liquid is then drained from the porous medium, allowed to remain without aeration beyond renewal of the consumed oxygen, and the operations repeated until vinegar is formed.

B. FULLMAN.

Destructive distillation of vinasses (U.S.P. 1,609,712).—See II.

XIX.—FOODS.

Determination of lactose in bread. M. W. F. SNETHLAGE (Chem. Weekblad, 1926, **23**, 578—580).—The aqueous extract is fermented with a pure culture of *Saccharomyces cerevisiæ* at 30°; the fermentation destroys all sugars other than lactose, which can then be determined by means of Fehling's solution.

S. I. LEVY.

Determination of benzoic acid in egg yolk. E. WALTZINGER (Chem.-Ztg., 1926, **50**, 949).—To 10 g. of egg yolk in 400 c.c. of water are added 42 c.c. of Fehling's copper sulphate solution, followed by 16 c.c. of *N*-sodium hydroxide solution, when the liquid should be weakly acid. After heating on the water bath at 80—90° to coagulate the precipitate, the whole is cooled, made up to 500 c.c., and filtered. 250 c.c. of the filtrate are rendered distinctly alkaline, evaporated to 20 c.c., filtered from the precipitated copper oxide, the filtrate is acidified with hydrochloric acid, and the benzoic acid dissolved by the addition of 40 c.c. of ether. 30 c.c. of light petroleum are then added, and after shaking the lighter layer is separated, the extraction with the mixed solvent being repeated. After washing the combined extracts with water, the solvents are distilled off and the benzoic acid remaining is dried at 50—60° to constant weight, dissolved in neutral alcohol, and titrated with 0·1*N*-alkali and phenolphthalein.

F. R. ENNOS.

Detection of apple in jams. C. F. MUTTELET (Ann. Falsif., 1926, **19**, 580—585).—The juices of preserved apples, pears, and quinces contain an excess of lævulose over dextrose, but dextrose predominates in the juices of apricots, peaches, greengages, and blackberries. In the case of red and white gooseberries, cassia, and strawberries, the two sugars are found in practically the same proportions. These facts apply equally to the fruit jellies. Apple juice may be detected in the jellies of citric acid fruits either by a determination of the sugars or by examining for malic acid; in jams of malic acid fruits, the malic acid or sugars may be determined. For apple jellies these determinations will give useful information of adulteration with pectins or pectic juices. The proportions of lævulose and dextrose in home-made jellies and jams, and in commercial "pure fruit" preparations, are tabulated.

D. G. HEWER.

Distinction between malt-coffee and cereal-coffee by means of the maltol reaction. T. MERL (Z. Unters. Lebensm., 1926, **52**, 321—323).—Coffee prepared from malted grain is distinguished by giving a positive maltol reaction, *i.e.*, the violet colour produced by treating a hot chloroform extract of coffee with ferric chloride solution.

A. G. POLLARD.

Black sausages in artificially-coloured skins. T. MERL (Z. Unters. Lebensm., 1926, **52**, 323—324).—Artificial colouring matter can be extracted from black-sausage skins by boiling with alcohol. After removal of the alcohol, potassium bisulphate produces a green colour in the extract. With concentrated hydrochloric acid an intense violet coloration develops.

A. G. POLLARD.

Volatile substances of foods. J. König (Chem.-Ztg., 1926, **50**, 992—994).—The gases evolved when various foodstuffs are heated at 100° for 2 hrs. in a stream of nitrogen have been examined. Carbon dioxide, formaldehyde, and higher aldehydes were evolved from all the foods. In addition, hydrogen sulphide was liberated from all the foods which contained albumin, hydrogen phosphide from milk and egg yolk, and methylmercaptan from cabbage and cauliflower. The relationships of the volatile substances to each other, and especially to the vitamin contents of the foods are discussed. Foods rich in vitamins evolved 2—10 times as much formaldehyde as those containing little or no vitamins. E. H. Sharples.

Formation of sucrose in potatoes during drying. de Wolff.—See XVII.

Patents.

Increasing the coagulating point of milk. W. O. Frohring (U.S.P. 1,609,617, 7.12.26. Appl., 15.6.26).—Caseinates of sodium, potassium, or calcium increase the coagulating point of milk and milk products, and are also desirable ingredients. They may either be added directly or substituted for a part of the protein content already present. F. R. Ennos.

Infant's food. C. Nielsen, Assr. to Abbott Laboratories (U.S.P. 1,607,844, 23.11.26. Appl., 24.3.22).—A product containing fat, carbohydrates, casein, and lactalbumin in approximately the same proportions by weight as in normal human milk is obtained by mixing cream, maltose, and dextrins, soluble lactalbumin, and orange juice with water. The mixture containing about 60% of solids is sprayed under a pressure of 100 lb./sq. in. through nozzles into a chamber heated by a current of air at 150—200°. The water is evaporated and the product precipitated as a fine powder. F. R. Ennos.

Method of pasteurising cheese. E. E. Eldredge (U.S.P. 1,607,064. 16.11.26. Appl., 16.2.25).—The cheese, after being ground, is mixed with a small quantity of water, together with 0·75% of ammonium tartrate and 0·25% of ammonium chloride. The whole is then heated by steam at 60°, and while melted is poured into containers to cool. F. R. Ennos.

Preparing fish for canning. H. R. Beard (E.P. 241,169, 31.8.25. Conv., 7.10.24).—The fish pass through a series of chambers in which they are subjected to rapid currents of heated air moving at 1500—2000 ft./min. parallel to the direction of movement of the fish. The temperature of the air in the first chamber in which the fish is partially dried is about 60°; in the second, where the cooking process takes place, it is 150—160°, and in the last or cooling chamber it is 15—20°. F. R. Ennos.

Food compound. S. L. Crawford (U.S.P. 1,605,219, 2.11.26. Appl., 20.10.20).—To a concentrated fruit flavour, from which the natural sugar and acid have been removed, pectin in the form of a syrup and an organic acid, such as tartaric or citric acid, are added. The mixture is then sterilised. F. R. Ennos.

Method of obtaining coffee flavour. J. L. Kellogg (U.S.P. 1,605,115, 2.11.26. Appl., 15.2.23).—After roasting and disintegrating the coffee bean, its essential oil, which retains the characteristic aroma of coffee, is expressed by pressure alone, the temperature being maintained below 65°. Only a small proportion of the caffeine is extracted, which is removable by centrifuging. F. R. Ennos.

Production of an article of food. H. Bollmann, Assr. to M. F. Foster (U.S.P. 1,606,052, 9.11.26. Appl., 28.5.25. Conv., 30.3.25).—Oil fruits are extracted with alcohol and benzene, the solvent is vaporised by steam, and the oil removed from the extract by washing with acetone. A wax-like mass of lecithin and phosphatides is obtained, which is mixed with chalk to form a non-agglomerating powder. F. R. Ennos.

Process and apparatus for treating raisins or other dried fruit. E. C. R. Marks. From Sun-Maid Raisin Growers of California (E.P. 262,988—9, [A] 16.2.26, [B] 17.2.26).—(A) The raisins, while subjected to the action of superheated aqueous vapour of low humidity at atmospheric pressure, are superficially heated for a short period at about 430°, then pass through a seeding machine, and are subsequently cooled in stages, first slowly and finally rapidly (cf. E.P. 262,990, *infra*), after which they are aerated to bring them to a uniform temperature. During cooling the raisins are subjected to a blast of atomised oil extracted from the seeds of the fruit. (B) The apparatus consists of two concentric, slightly inclined, non-communicating cylinders with pipes passing through the intervening space, by means of which aqueous vapour heated at 110—120° at atmospheric pressure is admitted to the central cylinder. The fruit, which is moved rapidly through the central cylinder by a spiral conveyor, is heated by gas at about 430°, circulating in the space between the two cylinders. F. R. Ennos.

Apparatus for conditioning heated raisins or other dried fruit. E. C. R. Marks. From Sun-Maid Raisin Growers of California (E.P. 262,990, 17.2.26).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Technical production of concentrated acetic acid. H. Suida (Oesterr. Chem.-Ztg., 1927, **30**, 1—5).—A lecture. The economics of five processes and the physico-chemical principles on which they are based are discussed, and it is shown that the author's processes (cf. B., 1925, 827, 899) have the cheapest working costs for the production of 98—100% acetic acid from 10% acid. Partition coefficients of acetic acid between water and 13 solvents and temperature–composition curves of the liquid and vapour phases of mixtures of acetic acid and water containing a water-insoluble solvent are given. E. H. Sharples.

Determination of nicotine in tobacco. R. R. T. Young (Analyst, 1927, **52**, 15—25).—The nicotine content is determined by a modification of Keller's method; 6 g. of dried and powdered material are shaken for 15 min. with a mixture of 100 c.c. of light petroleum, 20 c.c. of ether, and 10 c.c. of 20% potassium hydroxide solution, and after a further 15 min. 100 c.c. of the ethereal solution are filtered off, to which are added 10 c.c. of alcohol, 10 c.c. of water, and a few drops of dilute cochineal solution. The nicotine and cochineal pass into the aqueous liquid by shaking; a slight

excess of 0·1*N*-hydrochloric acid is added, and the excess titrated with 0·1*N*-alkali solution. 1 c.c. of acid = 0·0162 g. of nicotine. If a determination of ammonia is desired the above solution is run into a separating funnel, the last traces are washed in with 50 c.c. of water containing 15 c.c. of *N*-hydrochloric acid, and, after shaking, the lower aqueous layer is run off, and 150 c.c. of an iodine solution containing 50 g. of iodine and 100 g. of potassium iodide per litre are added. After some hours the solution is filtered, the filter paper washed with potassium iodide solution, and the liquid decolorised by sodium thiosulphate and distilled with sodium hydroxide into 25 c.c. of 0·1*N*-acid; the acid is back-titrated with 0·1*N*-alkali solution. The percentage of ammonia in insecticides does not appreciably affect results for nicotine obtained by the above method.

D. G. HEWER.

Examination of cantharides and its preparations. New cantharides reaction. L. DÁVID (Pharm. Ztg., 1927, **72**, 56).—1 g. of coarsely powdered Spanish fly is shaken violently for 5 min. with 15 c.c. of chloroform and 2—3 c.c. of aqueous ammonia. The chloroform extract is filtered off, dried with 0·5 g. of anhydrous sodium sulphate, filtered again, and evaporated on the water bath. 7 drops of fuming nitric acid are added, and the mixture is evaporated to dryness. To the warm residue are added 5—6 drops of a solution of 1 g. of vanillin in 100 c.c. of fuming hydrochloric acid, and the mixture is warmed on the water bath. The presence of cantharidin is indicated (usually after a few secs.) by the formation of an orange-red to brown-red colour. The reaction is negative with old samples of Spanish fly. Plasters or ointments are freed from fat by extraction with light petroleum (*d* 0·660) and the residue is examined as above.

E. H. SHARPLES.

Technical preparation of the silver compounds of the German pharmacopœia. F. CHEMNITIUS (Chem.-Ztg., 1926, **50**, 958—961).—*Argentum nitricum* (fused silver nitrate) is prepared by dissolving the metal in nitric acid (*d* 1·40) free from halogens in a 30-litre covered porcelain vessel with a copper steam jacket. Distilled water is added from time to time to moderate the reaction, which requires 24 hrs., the acid being added slowly. The solution is removed with porcelain ladles, filtered through paper, and evaporated in porcelain basins of 7—8 litres capacity over a steam-bath. The concentrated solution is inoculated with a silver nitrate crystal carefully covered with 1 cm. of distilled water and allowed to crystallise. The crystals are washed with distilled water and recrystallised once. If 90% (cupreous) silver has been used, three crystallisations are necessary. Mother-liquors are collected and in turn evaporated. From "100%" silver bismuth gradually accumulates and must finally be precipitated by dilution. From a copper mother-liquor silver is precipitated as chloride, and reduced by fusion with 2 pts. of calcined soda, the molten silver being poured into water to remove impurities. Gold may pay for recovery. The silver nitrate is fused with addition of ¼% of potassium nitrate and poured into silver-plated iron moulds. The yield of the process should reach 99·5%. *Argentum colloidale.* 10 kg. of silver nitrate are dissolved in concentrated ammonia, diluted to 20 litres, and 2·6 kg. of protalbinic acid (prepared from casein) diluted to the same volume. The solutions are then mixed, 20 litres of sodium hydroxide solution added, and the mixture is warmed to 65—70° with vigorous stirring. The temperature must not exceed 80°. When the reaction is complete the cooled solution is dialysed through a parchment membrane into distilled water. This requires 4—5 days, and the outer solution should be only slightly alkaline on completion. The silver solution is decomposed with ammonia and filtered after 24 hrs. The precipitate is dried on shallow porcelain basins at 35—40°. The product contains 75—78% of silver, and the yield is 98% of the theoretical. *Argentum proteinicum*, an albumin preparation containing 8% of silver. A syrup is prepared by hydrolysing gelatin in an autoclave, treating with sodium hydroxide and sodium hydrogen carbonate, filtering, and evaporating. The syrup is treated with silver nitrate solution in the required quantity, in a jacketed, enamelled vessel. After the first precipitate has redissolved sodium hydroxide is added, producing a brown extract soluble in water. This is bleached with hydrogen peroxide, and the mass dried on aluminium sheets *in vacuo*. Full working details for all these preparations are given.

C. IRWIN.

Simple method for the detection of adulteration of essential oils with alcohol. L. DÁVID (Pharm. Ztg., 1927, **72**, 56).—2 c.c. of the oil are shaken with 3 c.c. of water, and after settling the water layer is filtered off. 2—3 c.c. of a solution of 2 g. of molybdic acid in 100 c.c. of concentrated sulphuric acid are then added to the water to form a layer underneath. In the presence of small amounts of alcohol a blue ring is formed at the junction of the two layers after some hours. With large amounts an opaque blue ring is formed immediately, and the surface in contact with the acid has a dark grass-green colour. Oils containing aldehydes and alcohols are first extracted with light petroleum, and the water extract of the ether extract is used for the test. Many essential oils contain water-soluble constituents which form a coloured ring with the reagent, but a blue ring is given only by alcohol. The test may be used for the detection of alcohol in fatty oils, ether, chloroform, and other water-insoluble substances. 0·116% of alcohol can be detected.

E. H. SHARPLES.

Solubility of alkaloids in oils. G. TELLERA (Boll. Chim. farm., 1926, **65**, 737—740).—The use of oil and oleic acid as a solvent for alkaloids to be administered subcutaneously is discussed, and the proportions of oleic acid necessary to give neutral oleates of the alkaloids are given.

T. H. POPE.

Determination of benzoic acid in egg yolk. WALTZINGER.—See XIX.

PATENTS.

Manufacture of methyl alcohol. H. DREYFUS (E.P. 262,494, 13.6.25).—Methyl alcohol is formed when mixtures of or containing about 1 mol. of hydrogen or slightly less and 1 mol. of carbon monoxide are heated at not above 350—450° (preferably 200—300°) under pressures up to about 200 atm. (generally 50—150), in the presence of zinc oxide. The mixture may be produced from the separate gases, or water-gas or other industrial gases of similar composition may be used. If the gases

are passed through the catalyst at a relatively low speed, hydrocarbons or higher alcohols may be formed.
B. Fullman.

Catalysts for synthetic methyl alcohol production. J. C. Woodruff and G. Bloomfield, Assrs. to Commercial Solvents Corp. (U.S.P. 1,608,643, 30.11.26, and 1,609,593, 7.12.26. Appl., 26.5.26).—The catalyst consists of ferric hydroxide and (A) zinc oxide, (B) magnesium oxide. B. Fullman.

Production of formic acid. W. C. Arsem, Assr. to Commercial Solvents Corp. (U.S.P. 1,606,394, 9.11.26. Appl., 20.6.25).—Carbon monoxide and water react under pressure in the presence of a cuprous halide catalyst.
B. Fullman.

Manufacture of acetic acid. H. Dreyfus (E.P. 262,832, 13.6.25).—Approximately equimolar mixtures of hydrogen and carbon monoxide yield acetic acid when heated at 350—450° (preferably 200—300°), at pressures up to about 200 atm. (generally 50—150) in the presence of substances capable of forming acetates which decompose with the formation of acetic acid at 400—450° (preferably 200—300°). When these substances are themselves capable of effecting the combination of carbon monoxide and hydrogen, they may be used alone; otherwise they must be used together with such substances as are employed in combining these gases to form oxygenated organic compounds, other than zinc oxide. Catalysts which may be used are oxide of tin, copper, etc., copper acetate, methoxides of aluminium, etc., either alone or mixed with one another or with potassium or sodium acetate or methoxide etc.
B. Fullman.

Production of keten. H. Dreyfus (E.P. 262,364, 13.6.25).—Mixtures of approximately 3 mols. of carbon monoxide to 1 mol. of hydrogen yield keten when heated at about 400—450° (preferably 200—300°) under pressures of about 200 atm. (generally 50—150), in the presence of catalysts which can effect the combination of the gases to form oxygenated organic compounds. Such catalysts are zinc or copper oxide or chromate, copper, zinc, or tin with potassium or sodium carbonate or acetate, etc. The keten may be used directly for the production of acetic acid etc., or removed by cooling and liquefaction. B. Fullman.

Process for producing thiourea. F. S. Washburn (U.S.P. 1,607,326, 16.11.26. Appl., 5.6.20).—Crude calcium cyanamide is dissolved in water, the temperature being kept sufficiently low to prevent substantial decomposition; the insoluble impurities are removed and the solution is treated with a solution containing ammonium sulphide and dissolved sulphur.
D. Woodroffe.

Production of arsinic acids of aromatic compounds. A. J. Ransford. From L. Casella & Co., G.m.b.H. (E.P. 261,133, 2.9.25).—4-Nitro-2-aminophenol or its substituted derivatives (obtained by partial reduction of 2 : 4-dinitrophenols) yields 4-*nitro*-1 : 2-*dihydrobenzoxazolone*, m.p. 228—229°, or its substituted derivatives on treatment, in solution in alkali, with phosgene. From the latter nitro-compounds the corresponding arsinic acids are obtained by reduction etc. The following are described: 4-*amino*-1 : 2-*dihydrobenzoxazolone*; 1 : 2-*dihydrobenzoxazolone-4-arsinic acid*; 4-*nitro*-6-*methyl*-1 : 2-*dihydrobenzoxazolone*, m.p. 236—237°, and the corresponding *amino-compound*; 6-*methyl*-1 : 2-*dihydrobenzoxazolone-4-arsinic acid*; 6-*chloro*-4-*nitro*-1 : 2-*dihydrobenzoxazolone*, m.p. 210—211°, and the corresponding *amino-compound*; and 6-*chloro*-1 : 2-*dihydrobenzoxazolone-4-arsinic acid.* B. Fullman.

Manufacture of new complex antimony compounds. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 262,301, 15.2.26).—Antimonyl derivatives of polyhydric phenols having two adjacent hydroxyl groups (or their substitution products) react with neutral salts of thioglycollic acid, yielding complex antimony compounds. Examples are given of the preparation of pyrocatechylantimonyl, pyrogallylantimonyl, and gallylantimonyl *derivatives* of thioglycollic acid. The substances have therapeutic application. B. Fullman.

Manufacture of dixanthogen. W. Hirschkind, Assr. to Great Western Electro-Chemical Co. (U.S.P. 1,606,573, 9.11.26. Appl., 24.5.26).—A solution of an alkali xanthate is electrolysed until a part is converted into dixanthogen. Alcohol and carbon disulphide are added, and when the free alkali has disappeared the electrolysis is repeated. B. Fullman.

Manufacture of phosphoric esters of carbohydrates and polyvalent alcohols. Soc. Chim. des Usines du Rhône (E.P. 251,229, 7.9.25. Conv., 27.4.25). —See U.S.P. 1,598,370; B., 1926, 964.

Manufacture of derivatives of cellulose and formic acid (E.P. 260,650).—See V.

[Compound for] wood preservation (U.S.P. 1,612,102).—See IX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Sensitisation of photographic plates. W. Leszynski (Z. wiss. Phot., 1926, **24**, 261—275).—The "absolute" sensitivity to blue, green, and red light was measured on an unsensitised pure silver bromide emulsion, and the same emulsion sensitised to green and red light. The amount of the "primary" separated silver in an Erythrosin-bathed emulsion exposed to green light was measured by the method of Eggert and Noddack (Z. Physik, 1923, **20**, 299) and related to the number of quanta incident in exposure. From these and other experiments it is concluded that the ratio of the "primary" separated silver on the surface and inside the grains is the same for blue and green exposure. The number of silver atoms formed on exposure of an Erythrosin-bathed plate to green light is at least 20 times as great as the total number of the sensitising dyestuff molecules present. The following explanation is given for sensitisation with Erythrosin. The dye adsorbed to the surface of the silver bromide grain is responsible for the primary light absorption, which results in the transfer of an electron from a bromine ion to a silver ion. The presence of silver nuclei in the interior of a grain is supposed to be due to the freed electron being able to wander to a distant silver ion. W. Clark.

Sensitivity of photographic emulsions. II. Hydrogen-ion concentration and the silver bromide-thiocarbamide complexes. S. O. Rawling (Phot. J.,

1926, **67**, 42—48; cf. B., 1926, 1029).—Emulsions prepared from a normal gelatin, and from a sample of the same gelatin which had previously been used in the manufacture of an emulsion and then freed from the silver halide by centrifuging, gave speeds of the same order. The emulsion made from the centrifuged gelatin also showed a very marked increase of speed with increase in hydroxyl-ion concentration of digestion. The speed of an emulsion which has been digested for some time is almost instantly lowered by the addition of acid, and by bringing the emulsion back to its original hydrogen-ion concentration it is just as rapidly restored. These changes cannot be due to the presence of a greater or less amount of silver sulphide derived from the silver bromide–thiocarbamide complexes according as the emulsion is more or less alkaline. The sudden changes of speed brought about by such changes in hydrogen-ion concentration probably have their origin in a mechanism other than that which brings about the slow changes in photographic properties normally observed throughout the digestion of emulsions. W. Clark.

Chemical aspect of sulphide sensitivity. K. C. D. Hickman (Phot. J., 1927, **67**, 34—41).—It is suggested that the specks of silver sulphide, to which are ascribed the sensitivity of a high-speed silver bromide emulsion, play a chemical part during exposure by acting as halogen absorbers, the reaction being of a secondary order and not involving optical sensitisation. Evidence is given indicating that silver sulphide deposited on the grains of an unexposed photographic emulsion is oxidised by exposure of the emulsion to light to give a print-out image, and an increased yield of metallic silver is produced. In a study of the reaction on the ordinary laboratory scale, *i.e.*, the reaction between bromine and silver sulphide, no liberation of metallic silver was observed. It is considered probable, however, that on the scale obtaining in latent image formation the bromine liberated atom by atom from silver bromide in contact with excess of silver sulphide, during exposure, may liberate silver as metal. Speculations are made concerning certain consequences of the theory. W. Clark.

Patents.

Colour photography. V. Weil (U.S.P. 1,609,232, 30.11.26. Appl., 8.10.25. Conv., 23.8.23).—The light-sensitive layer is impressed with a multi-colour screen by mechanical printing with a transparent colouring material, arranged as points of colour separated by very small spaces through which the solutions used in preparing the prints can penetrate down to the absorbent sensitised layer. W. Clark.

XXII.—EXPLOSIVES; MATCHES.

Theory of explosive reaction. H. Muraour (Bull. Soc. chim., 1926, [iv], **39**, 1711—1716).—For every type of explosive molecule there exists a definite temperature of decomposition. Three ways of approaching this temperature are examined—slow heating of the explosive, ignition in a closed vessel, and detonation. On slowly heating a simple solid explosive (*e.g.*, lead azide) some gas is always evolved before the temperature of decomposition is reached, this probably being due to a kind of Maxwell distribution among the molecules of solid, some of which are therefore above the mean temperature. With other types of explosive (*e.g.*, nitroglycerin) the reaction may proceed differently according to the rate of heating, relatively slow heating giving partial decomposition of the molecule with formation of a non-explosive residue, and faster heating giving total decomposition at once. When the powder ignites in a closed vessel under pressure, the gaseous molecules evolved decompose the unchanged molecules of explosive by the shock of collision, so that the velocity of ignition should be a function only of the pressure of the gas, and of the surface of the powder exposed. With most powders the latter quantity is indeterminate, but the experiments carried out by the authors on vitreous explosives, which possess a definite surface, support these views. In detonation the energy necessary for decomposition is supplied by the explosive wave. S. J. Gregg.

Patents.

Explosive. A. C. Scott (E.P. 262,491, 12.8.25).—The impregnation by nitro-compounds of cartridges containing a salt with readily available oxygen, *e.g.*, chlorate or perchlorate of potassium, or ammonium nitrate, is assisted by impregnating the cartridge with a liquid spreading agent, *e.g.*, kerosene, which enables the nitro-compound to become evenly disseminated through the mass of the salt. The weight of the spreading agent may be 1—4% by weight of the nitro-compound or may exceed 4%. Desensitising and/or cooling agents may be employed. S. Binning.

Manufacture of dinitrotoluene (E.P. 263,018).—See IV.

XXIII.—SANITATION; WATER PURIFICATION.

Head losses in the rapid sand filters at Cambridge, Mass. R. G. Tyler, W. A. Danielson, & M. Lebosquet, jun. (J. New Eng. Water Works Assoc., 1926, **40**, 322—344).—Tests made during actual filtering operations indicate that head losses due to the friction in the sand itself are relatively unimportant when compared with those due to clogging and to the floc film on the filter surface. The effective filtering stratum consists of the floc layer on the top of the sand and the fine sand which supports it. With coarse floc, removal is by surface retention; with fine floc, the filter functions more as a sedimentation basin with greater removal in the voids. A greater percentage of suspended solids is removed on the surface of the filter with aluminium sulphate floc than with the floc of sodium aluminate. W. T. Lockett.

Determination of nicotine. Young.—See XX.

Treatment of water for use in the manufacture of artificial silk. Brysilka Ltd., and F. W. Schubert (E.P. 262,369, 4.8.25).—Water is fed into a de-aerating chamber in which a vacuum is maintained, the dissolved air being thus liberated and part of the liquid vaporised with consequent cooling of the system. The air and vapour are drawn off, and alternately compressed to raise the temperature and cooled to condense the steam in their passage through a series of ejectors and intercoolers, the condensed water being returned to the de-aerating vessel, while the liberated air and steam residue are blown to waste. B. P. Ridge.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 4, 1927.

I.—GENERAL; PLANT; MACHINERY.

Method of measuring the size of particles. P. Lukirsky and M. Kosman (J.S.C.I., 1927, 47, 21—25 t).—A method for the study of the distribution of particles with respect to their sizes is described, the hydrostatic pressure at one or in two points of the falling suspension of powder being determined at different moments. A method has been worked out for the measurement of the difference of levels due to those pressures. The method affords a simple means of measuring this difference with an accuracy up to 1 micron.

Particle size and distribution by sedimentation. J. H. Calbeck and H. R. Harner (Ind. Eng. Chem., 1927, 19, 58—61).—The authors describe the experimental details of the determination of sedimentation gravimetrically, which they prefer to centrifugal and "change of density" methods. The derivation of sedimentation curves and the development therefrom of distribution curves and their rectangular equivalents —"histograms"—are explained from first principles and illustrated from the results of experiments with samples of red lead and litharge. S. S. Woolf.

Action of acids and alkalis on fuller's earth. F. Weldes (Z. angew. Chem., 1927, 40, 79—82).—Improvement in the bleaching properties of fuller's earth by preliminary treatment with acids is not due to hydration of the silica, but to solution of part of the alumina and iron oxide, principally the former, resulting in a highly porous product with an extensive active surface. The use of hydrochloric or sulphuric acid is essential to attain the maximum effect, as the hydrogen-ion concentration of organic acids, such as tartaric acid, is insufficiently high to dissolve out the necessary quantity of the oxides. Dilute alkalis impair the bleaching properties by forming silicates within the pores, but the activity can be restored by subsequent treatment with dilute inorganic or organic acids; prolonged treatment with strong alkalis causes solution of considerable quantities of the silica, destroying the structure of the material and thereby rendering it inactive and incapable of further activation.
L. A. Coles.

Calibration of the burette consistometer. W. H. Herschel and R. Bulkley (Ind. Eng. Chem., 1927, 19, 134—139).—Flow-pressure data on translucent and not too stiff materials may be obtained by using a simplified form of Auerbach's burette consistometer. Two such instruments were calibrated by flow tests on standard liquids, corrections to the ordinary expression for stream-line flow of a viscous liquid being derived to allow for (*a*) surface tension effects and (*b*) variations in the coefficient of the kinetic energy correction.
S. S. Woolf.

Device for adjusting the levelling bulb on a gas-analysing apparatus. A. M. McCollister and C. R. Wagner (Ind. Eng. Chem., 1927, 19, 86).—Accurate adjustment of a levelling bulb may be brought about by using a sliding block for carrying the ring for the bulb, actuated by a screw fitted with a knurled wheel (or rack and pinion). The device is carried on a ring stand clamped for rough adjustment. An ordinary split ring may be cut in two about the middle of the shank, and both ends threaded, and the sliding block drilled and tapped to receive the ring, and the main part fitted with the clamp portion of the original ring.
D. G. Hewer.

Mixing and agitation. M. Pailly (Rev. gén. Colloid., 1926, 4, 295—298, 328—333).—A brief description of known methods and apparatus.

Patents.

Apparatus for determining the moisture content of masses of particles [*e.g.*, moulding sand]. R. L. McIlvaine (E.P. 263,450, 8.9.26).—A tube enclosing an electric battery in circuit with an indicating instrument is provided with a plunger of insulating material carrying a pair of terminals and slidable in the open end of the tube against spring pressure. Means are provided for holding the plunger rigid with the tube until the open end of the tube has been inserted into the particles. The plunger is then free to slide inwards to close a switch which completes the indicating circuit through the terminals and the particles. H. Holmes.

Apparatus for determining the specific gravity of gases, in which a stream of gas flows in a tube provided with a baffle. J. H. Reineke (G.P. 432,520, 25.3.24).—Gas flows at a constant rate from slits in two opposed vessels connected with a gas meter and clockwork rotating in a case, and the specific gravity is determined from the law of effusion of gases through an orifice. J. S. G. Thomas.

Feeding solids into or removing solids from vessels under pressure. J. Y. Johnson. From Badische Anilin u. Soda Fabrik (E.P. 262,901, 5.11.25).—The powdered or granular material is forced into the vessel against a pressure of 100 atm. or more through a tube in which it forms a gas-tight closure. The material entering the tube from a hopper is pushed forward by a reciprocating plunger, and if its caking quality is insufficient to make the closure gas-tight, a layer of rubber, paper, cardboard, metal, or other material capable of providing such a closure is introduced at

each forward stroke of the plunger. A plunger device may be used similarly for pressing material out of the vessel through a tube. H. HOLMES.

Dispersing substances in water. W. B. PRATT, Assr. to RESEARCH INC. (U.S.P. 1,609,308, 7.12.26. Appl., 13.11.24).—Gums, waxes, asphalt, or like semi-solid substances are agitated with water (10—15%) between 0° and 100° until the liquid is completely absorbed, a dispersing agent (4%), such as clay, agar, or glue, is added, and the mass is then mixed with increasing amounts of water, until dispersion occurs. T. S. WHEELER.

Producer or shaft furnace. WOODALL-DUCKHAM (1920), LTD., and J. W. REBER (E.P. 262,668, 7.7.26).—The cooling jacket is constructed in upper and lower sections supplied with water under high and low pressures respectively, and the blast nozzles extend through the lower section. The water circulates in the lower section in a closed cycle, including a heat exchanger in which the water passing through the upper section is heated. H. HOLMES.

Centrifugal separators for liquids. F. GRIMBLE, M. N. CAIRD, and E. COOMBS (E.P. 262,902, 7.11.25).—A centrifugal separator is provided with a filtering medium placed in the path of the liquid flowing from the outer annular space to the neck of the bowl. The filter material may be placed between the usual set of conical separating plates, or made up to an appreciable depth to take the place of some of the separating cones. An additional filter comprising a medium sandwiched between two perforated plates may be placed at the neck or overflow weir of the bowl. B. M. VENABLES.

Centrifugal separators or clarifiers. F. MORTENSEN (E.P. 263,021, 18.5.26).—Inside the bowl of a centrifuge are placed one or more inverted cups or bells, which are screwed down to make liquid-tight joints at their lower and wider ends. Heavy matter is then separated on the interior surfaces of the bells as well as on that of the outer bowl, the transfer ports or passages from bell to bell being arranged as near the axis as possible. One or more of the bells may be arranged to have a permeable filter surface. B. M. VENABLES.

Separators. FULLERTON, HODGART, AND BARCLAY, LTD., and J. C. ASTON (E.P. 263,284, 3.12.25).—Liquid from which it is desired to remove solid matter is admitted at high velocity tangentially to the lower part of a cylindrical vessel, and the cleaned liquid exhausted tangentially from the upper part. The solid matter moves to the centre of rotation and drops through a central hole to the space between the double bottoms of the vessel, where it settles. B. M. VENABLES.

Centrifugal separators. P. FESCA (C. A. FESCA & SOHN) (E.P. 263,348, 3.3.26).—A centrifugal separator of the type having two pans or bowls placed base to base, which are drawn apart to discharge collected solid matter, is arranged with both pans perforated, the upper one rotating but fixed axially, the lower one rotating and movable axially by hydraulic or pneumatic means. B. M. VENABLES.

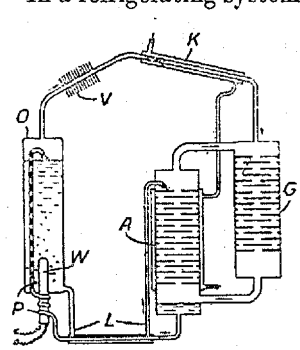
Drums for centrifugal separating apparatus. K. J. SVENSSON and K. A. P. NORLING (E.P. 263,443, 3.9.26. Conv., 29.12.25).—A centrifugal separator for material containing at least two liquid constituents, also solid matter in suspension, is arranged so that the finer solid matter (slime) is removed continuously from the bowl along with the heavier liquid constituent, thus preventing or postponing the necessity of stopping the bowl for cleaning. This is effected by making the discharge passages for heavy liquid leading from the outer part of the bowl to the discharge neck small, and thus maintaining the velocity of the outflowing heavy liquid high enough to carry the slime with it. The position of the entrances to these passages will vary with the nature of the solid matter in the feed; if the solid matter contains no pieces large enough to block the passages, they may start at the extreme outer part of the bowl, but if chips or large pieces are present, the passages must start rather inwards, so as to allow space for large pieces to collect. B. M. VENABLES.

Hydro-extractors. BAKER PERKINS, LTD., and J. E. POINTON (E.P. 263,303, 22.12.25).—To reduce the power demand for operation of two or more extractors, these are mounted on a common base with a single driving motor, and the transmission mechanism is provided with clutches enabling the extractors to be started into operation successively, and to be maintained in operation simultaneously. H. HOLMES.

Jaws or breaker plates for stone crushers and the like. P. BECHGAARD (E.P. 263,070, 12.10.26).—Jaws for crushers with corrugations or other form of projections on them are constructed with cavities within the substance of the jaw which are of the same form as and immediately behind the outer corrugations. When the original outer ridges are worn away the cavities are exposed and form fresh corrugations. The cavities may either be left empty or filled (during or after the casting of the jaw) with softer or more brittle metal, which is subsequently driven out. B. M. VENABLES.

Crusher. R. C. GREENFIELD, Assr. to ALLIS-CHALMERS MANUF. Co. (U.S.P. 1,607,615, 23.11.26 Appl. 30.9.22).—A crusher comprises a conical dish, in which rotates a vertical shaft carrying two conical crushing heads, so that there are formed two tapering crushing spaces, one above the other. Packing of the material in the crusher is avoided. T. S. WHEELER.

Absorption refrigerating apparatus. A. L. MOND. From PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (E.P. 262,870, 19.9.25).—In a refrigerating system employing, *e.g.*, ammonia, water, and hydrogen, arranged as represented in the diagram, where V is a rectifier, K a condenser, G an evaporator (or refrigerator proper), A an absorber, and O a boiler, the invention lies in the arrangement of the conduit, P, which carries liquid saturated with absorbent from the absorber, A, to the boiler, O. The



saturated liquor, after pre-heating by heat exchange with the returning barren liquor in the conduit, L, is

boiled by a heater, W, within the convolutions of the conduit, P, and the bubbles of gas thus produced alternate with short columns of liquid in the vertical part of the conduit, P, which is kept small for the purpose of preventing free exit of the gas bubbles. The gas-lift pump effect produces a circulation throughout the boiler, absorber, and conduits, P and L. The liquid is further boiled by the same heater, W, extending into the boiler, O. B. M. VENABLES.

Refrigerating apparatus. W. H. CARRIER, and CARRIER ENGINEERING CORP. (E.P. 263,052 and 263,206, 11.8.25).—(A) In a refrigerating system utilising a volatile liquid as refrigerant, and comprising a main evaporator (or refrigerator), a compressor and a condenser, a portion of the uncondensed vapour and non-condensible gases is withdrawn from the top of the main condenser (which is preferably cooled by a downward counter-current flow of water) and led through the heating coil of a rectifier (see below) to an auxiliary condenser and separating chamber, which is cooled by cold refrigerant drawn from the main evaporator. From this separating chamber the non-condensible gases are pumped to atmosphere, and the condensed refrigerant joins the main bulk of liquid in the evaporator. The rectifier is supplied with a portion of liquid from the spraying device at the top of the evaporator, which is separated into impurities (drawn off at intervals) and pure vapour of refrigerant, which re-enters the evaporator. (B) An approximately constant rate of rectification is maintained by means of a heating coil, which is only partly submerged in a new charge of comparatively pure and easily-boiling liquid, but fully submerged in the liquid when it contains practically nothing but impurities. B. M. VENABLES.

Apparatus for heating liquids [water]. A. G. TAYLOR and H. STUBBING (E.P. 263,401, 4.6.26).—The liquid is circulated through a casing traversed by tubes through which combustion gases from a heater pass upwards to an outlet flue. The top of the casing is convex, and a baffle, slightly spaced from it above the tubes, is similarly shaped. The bottom of the casing is concave, and is removably mounted on a stand enclosing the heater. The outlet flue is carried centrally by a cover removably mounted on the top of the casing. H. HOLMES.

Fractional distillation. R. B. CHILLAS, JUN., Assr. to ATLANTIC REFINING Co. (U.S.P. 1,612,572, 28.12.26. Appl., 30.6.25).—A proportion of the total descending reflux liquid is removed at a predetermined point, and this proportion is maintained constant irrespective of variations in the rate of the reflux flow. H. HOLMES.

Apparatus for separating or filtering liquids from materials containing them. LEVER BROS., LTD., and J. L. CLOUDSLEY (E.P. 262,840, 11.8.25).—The material is compressed within a cylinder by a worm on an axial shaft, and the expressed liquid is discharged between the coils of a helix of stout wire or tubing constituting a liner for the cylinder. The coils may be grooved or notched to expedite the discharge, and the inner face of the cylinder or of a plain liner therein may be provided with conduits for the further discharge of the liquid from the apparatus. When a tubular helix is used a heating or cooling fluid may be circulated through it, or a fluid escaping through perforations in contact with the material may be supplied. H. HOLMES.

[Boiler] furnaces. T. E. ROBERTSON. From POWER SPECIALTY Co. (E.P. 263,369, 10.4.26).

Dialysing membranes (U.S.P. 1,608,886).—See V.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Lignin and oxycellulose theory [of coal formation]. J. MARCUSSON (Z. angew. Chem., 1927, **40**, 48—50; cf. B., 1926, 809).—Completely decayed wood, three samples of peat, a lignite, and a brown coal have been submitted to the method of analysis previously described, with results confirming the author's theory of coal formation. The decayed wood gave a high yield (17·7%) of non-volatile water-soluble acids, consisting partly of glycuronic acid and partly of acids which appear to be transition products between glycuronic and humic acids. The cellulose content of the peat decreased with the age of the sample, being replaced by oxycellulose and humic acids; no progressive increase was observed in the lignin content. Similar results were found for the lignite and brown coal. The peat analyses of Odén and Lindberg (B., 1926, 568) are open to criticism. Extraction by sulphurous acid under pressure will remove the water-soluble acids with the lignin, giving much too high a figure for the latter, and will moreover attack the cellulose. The author's results are confirmed by those of Keppeler (B., 1920, 394 A). A. B. MANNING.

Cleaning of small coal. A. GROUNDS (Proc. S. Wales Inst. Eng., 1927, **42**, 529—577).—Various types of vibrating screens (Overstrom, Sturtevant, Hum-mer) and concentrating tables (H. H. Overstrom, Massco, Butchart, H.H. Universal Curvilinear) used for coal washing are described, some of them in detail, and a number of results of recent tests quoted. Electrically operated screens require the installation of special generators, and compare unfavourably, for example, with the Overstrom, of which the mechanism, based on the vibrations of an unbalanced pulley, is extremely simple. Concentration tables differ from one another mainly in the distribution of the riffles. It has been found possible to replace the usual linoleum covering for the table by rubber, the riffles being rubber covered also; the life of the covering is thereby considerably increased. Systems depending on air separation, in particular the Birtley table, are described. The advantages of air separation, at any rate for coal above $\frac{1}{16}$-in. size, are more than offset by the added screening capacity required, the extra power absorbed by the fans, and the added capital expenditure for dust collecting plant. The question of de-watering is dealt with, descriptions being given of the H.H. machine, the Carpenter centrifuge, the Oliver rotary filter, and several types of coal drier. A. B. MANNING.

Torbanite and its treatment by the Bergius method. A. W. NASH (J. Inst. Pet. Tech., 1926, **12**, 568—581).—The author reviews the researches which

have been made on the nature of torbanite found in different parts of the world, and traces its origin to matter similar to Coorongite, a product of decomposition of algæ found in lagoons in South Australia. Experiments were made in berginising New South Wales torbanite in presence of hydrogen or nitrogen, the results being compared with those of ordinary and steam distillation. A decrease of pressure was noted in the experiments with hydrogen, whereas an increase in pressure occurred with nitrogen. The yields of oil were inferior to those of ordinary distillation, both in the cases in which phenol was added as a disperse medium and without phenol. Prolongation of the treatment led to thermal decomposition of a part of the oils. The residual gases of hydrogenation contained 8·5—26·3% of methane. H. MOORE.

Investigation of the behaviour of solid fuels during oxidation. II. B. MOORE and F. S. SINNATT (Fuel, 1926, **5**, 377—380).—The changes in the properties of freshly-mined coal after 1 and $2\frac{1}{2}$ years' storage exposed to air were examined by the method previously described (B., 1925, 486). The glow-point temperature of the coal and the ignition temperature of the volatile matter were determined. The ignitions of the volatile matter in the coals after storage were less in magnitude than those of the freshly-mined samples, and the glow-point temperatures increased slightly during storage. The tendency of a coal to ignite is diminished by storage due to the formation of compounds of greater thermal stability, or with higher temperatures of ignition. With coals obtained from seams associated with "gob fires" the time interval between glow-point and ignition of the volatile matter is shorter than for other coals.

A. C. MONKHOUSE.

Oxidation of the constituents of a resinous Utah coal. J. D. DAVIS and D. A. REYNOLDS (Fuel, 1926, **5**, 405—411).—A non-coking Utah coal was extracted with benzene at 275° and 750 lb. pressure; the extract was separated into solid and oily bitumens using Fischer's method (B., 1925, 656). The rates of oxygen absorption at 60° over periods of 100 hrs. were determined for the three separated constituents, and the results compared with those of the original coal. The absorption of oxygen is shown to be due to the oxidation of all parts of the coal substance, and not to any particular constituent of the coal. The residue after extraction oxidised twice as rapidly as the original coal on account of the increased surface exposed. Similar results were obtained with the bitumens after being dispersed on pumice. Water is the main product of oxidation of the bitumens, carbon dioxide predominating in the oxidation of the residue. A. C. MONKHOUSE.

Thermochemical comparison of various types of lamp black. L. HOCK and S. BOSTROEM (Kautschuk, 1927, 21—22).—The heat generated on wetting amorphous carbon such as lamp black or gas black with benzene is believed to be inversely proportional to the radius or surface area of the constituent particles. The results place lamp black in an intermediate position between gas black and thermatomic black, and reveal differences between various blacks of the same type.

D. F. TWISS.

Comparison of vitreosil, illium-alloy, and platinum crucibles for determination of volatile matter in coal. H. M. COOPER and F. D. OSGOOD (Fuel, 1926, **5**, 381—385).—Crucibles of vitreosil, platinum, and illium-alloy (62·21 Ni-Cr, with Cu, Mo, W, Fe, Mn, and Al) are compared for the determination, by the standard American method, of the volatile matter of 10 fuels containing 1—48% of volatile matter. Vitreosil and illium-alloy crucibles give results in agreement with those obtained by using a platinum crucible, except with high-volatile non-coking coals. The disadvantage of the vitreosil and illium-alloy crucibles is their high sp. heat, and tests using a water-cooled plate gave times of cooling of 6—8 min. as compared with $2\frac{1}{2}$ min. for platinum crucibles. For ease of manipulation and long service platinum and illium-alloy are superior to vitreosil. A. C. MONKHOUSE.

Influence of ash on calorific power. R. STUMPER (Bull. Soc. chim. Belg., 1926, **35**, 417—421).—The net calorific power, P_N, calculated from the heat of combustion, H, determined by the bomb calorimeter, and the percentage of ash, a, by the formula $P_N = 100H/(100-a)$, is plotted against the ash content, a, for a large number of Belgian coals, and found to fall slightly as the value of a rises. The variation is only 0·4% of the mean value for values of a below 20, but becomes more considerable when a is over 20.

S. I. LEVY.

Trend of design in modern coke-oven construction and its bearing upon refractory materials. A. E. J. VICKERS and A. T. GREEN (Fuel, 1927, **6**, 4—14). —The systems of heating used in the principal types of coke oven are outlined. Recent work on the refractory properties of silica brick, siliceous firebrick, and fireclay is summarised in so far as it bears on the use of these materials in coke oven construction (cf. Wilson, B., 1924, 514; Dale, B., 1925, 717; Middleton, *ibid.*, 947; Green, B., 1926, 947). It is concluded that silica material has many advantages over fireclay; it stands up better under load, is more resistant to corrosion, and enables the ovens to be operated more quickly. A full bibliography is appended.

A. B. MANNING.

Residual and extinctive atmospheres of flames. T. F. E. RHEAD (Fuel, 1927, **6**, 37—40).—The composition of the residual and extinctive atmospheres of flames depends on (1) the relative rates of diffusion of the combustible gases, (2) their oxygen requirement, (3) the relative speed of flame propagation in the air–gas mixture, (4) the extent of the range of inflammable mixtures, and (5) the specific heat of the products of combustion as compared with that of the diluent gas in the original atmosphere. The low value of the oxygen content of the residual atmosphere from the combustion of hydrogen (5·6%) as compared with the corresponding values for methane (16·6%) and carbon monoxide (12·3%) are thus explained. The results obtained in determining these values are to some extent dependent on the experimental conditions. Some measurements of the oxygen content of atmospheres (oxygen–nitrogen mixtures) which just cause extinction of flames of methane, ethane, propane, and butane, have been made

by a "flow" method, in which a flame of the gas issuing from a jet was supplied with a steady stream of an "atmosphere" of which the oxygen content could be varied. The results were influenced by the size of orifice, the speed at which the gas issued, and the relative speed of the gas stream and the surrounding atmosphere; the values for methane, for example, varied from 16·6 to 18·2%. The results have a bearing on such problems as the sealing off of gob fires and the design of burners for domestic gas ovens. A. B. Manning.

Spectrography of flames in a combustion engine. A. Henne and G. L. Clark (Compt. rend., 1927, **184**, 26—28).—The explosion spectra of a combustion engine operating normally, with "knocking," and in the presence of anti-detonators, have been photographed at four stages of each explosion. The extent of the "knocking" is measurable by the movement of the lines of the normal spectrum towards the ultra-violet, and is greatest in the first quarter of the explosion. "Knocking" is due to a sudden liberation of energy, the effect of the addition of an anti-detonator being to distribute this over a longer period of time. All the substances used (lead tetraethyl, aniline, and ethylenic hydrocarbons) produced the same spectrum as was obtained under normal circumstances. Lines due to lead appeared only in the first quarter of the explosion, indicating that the catalytic action takes place at the beginning of the reaction. The artificial "knocking" was produced by pressure. J. Grant.

Addition of waste gases from industrial furnaces to a producer blast. C. Ammon (Chem.-Ztg., 1927, **51**, 41).—A considerable amount of steam has to be added to a producer blast to obtain a rich gas and to lower the temperature of the gasifying zone. The same results can be attained by using waste gases from furnaces, there being sufficient moisture for the production of carbon monoxide and hydrogen. The resulting producer gas has a smaller moisture content, which is advantageous for metallurgical processes. The small initial cost of the additional plant required is soon covered by the saving in steam. W. G. Carey.

Oxidation of ammonium sulphide. [Removal of sulphur from gas.] M. P. Applebey and J. A. Lanyon (J.C.S., 1926, 2983—2994).—The oxidation of the sulphur contents of coal gas without the preliminary removal of ammonia is attempted, and thus ammonium sulphate formed without the supply of extraneous sulphuric acid. Ammonium sulphide was quantitatively transformed into a mixture of ammonium sulphate and sulphite at 450—650° in the presence of catalysts. The optimum mixture is 650 vols. of air, 50 vols. of hydrogen sulphide, and 100 vols. of ammonia. Alumina, best in the form of gel, is the most efficient catalyst, but pumice can also be used. Ferric oxide and chromic oxide are unsuitable because they catalyse the oxidation of ammonia, hydrogen sulphide acting as a promoter to ferric oxide. The efficiency of the above changes is seriously interfered with by coal gas. The yield was considerably reduced and much contaminated with tar, especially at the lower temperatures; at higher temperatures a green liquid was formed with a strong garlic odour suggesting carbonyl sulphide. Experimental devices described include an apparatus for generation, storage, and delivery of pure hydrogen sulphide at constant pressure, a quantitative and rapid absorbent for hydrogen sulphide, and a method for the determination of sulphite and thiosulphate in a mixture of the two. W. Thomas.

Accuracy of the A.S.T.M. distillation method. Bataafsche Petroleum Maatschappij (J. Inst. Pet. Tech., 1926, **12**, 586—590).—Investigations of thermometer lag with thermometers of different types failed to show any constant difference, the results being irregular. Comparisons were made of the results obtained by four experimenters, carrying out five distillations each on four different pieces of apparatus. The smallest variation was in the successive results of one experimenter on the same apparatus, being 1° for initial and final b.p. and 0·2° for the middle distillation range. These errors become 1·3° and 0·4—0·5° when one observer works with various apparatus, and with various observers and apparatus 1·4° for initial b.p., more than 0·5° for middle range, and final b.p. 1·30—2·5°. The accuracy with which the volume of benzine distilling at a certain temperature is determined is equal to accuracy of temperature reading multiplied by cotangent of the angle of the distillation curve at that point. H. Moore.

Solvents for separating aromatic and aliphatic hydrocarbons in oils. O. Göhre (Petroleum, 1927, **23**, 73—77).—After a survey of the solvents previously used, the author discusses several new solvents that he investigated, including four which give satisfactory results. Lævulic acid, which can be used at the ordinary temperature, and is readily recovered by extraction with water, gives theoretical results for the aromatic content of oils containing up to 90% of these constituents. In extracting light oils or oils of medium viscosity the proportion of solvent to oil is $3\frac{1}{2}:1$, but with highly viscous oils or mixtures containing turpentine oils a proportion of 2 : 1 gives more reliable results. Phenylhydrazine is suitable for extracting many oils, the proportions varying with the nature of the oil, but it is not suitable in the case of hydrocarbons of low b.p. Glycol monoacetate in the proportion of 3 : 1 gives reliable results at the ordinary temperature for light oils and oils of medium viscosity, and of 2 : 1 for highly viscous oils, except where the aromatic content is high, when the mixture must be cooled with ice. In such cases lævulic acid is to be preferred. Furfuraldehyde used in the proportion of **1** : 1 is a reliable solvent for extracting all oils; it must, however, always be used at −10°. L. A. Coles.

Polymerisation. I. H. I. Waterman and J. N. J. Perquin. II. H. I. Waterman and J. C. Jamin (J. Inst. Pet. Tech., 1926, **12**, 506—509, 510—517).—I. Traces of oxygen may cause decomposition of hydrocarbons at a low temperature. Various unsaturated hydrocarbons were kept in contact with bleaching earth or active carbon, and their properties after distillation compared with those of the products of distillation of the untreated substances, the result being tabulated. Some hydrocarbons liberate heat on being mixed with the porous substance as in the case of hexylene and silica gel, but the product may not be changed, except when much heat is liberated. The

reaction with the porous substance is accelerated when the temperature is raised above 100°, as with indene and floridin at 140—150°. Silica gel caused only a slight change in amylene. The bromine number was often increased by distillation, but the results were irregular. Various hydro-aromatic and saturated hydrocarbons gave low bromine (addition) numbers. How far these were due to contamination of the substances examined could not be determined.

II. When 2 pts. of amylene were mixed with 1 pt. of norit, a rise in temperature was observed; after 28 hrs. the mixture was distilled at a pressure of 25 mm. of mercury, and the vapours were condensed with solid carbon dioxide and alcohol. The quantity of hydrocarbon remaining in the norit varied inversely with the moisture content of the latter, and could not be expelled by heating at 40° *in vacuo*. The addition of pumice stone caused the distillation temperature to be raised, and the residue to smell of turpentine. Amylene was treated with norit with about 1·6% moisture content for 24 hrs., and distilled under high vacuum. An examination of the amylene left in the norit showed that it had undergone little change, its unsaturation toward bromine having only increased 0·3%. Amylene was heated and its vapour passed over dried floridin in Lebedev and Filonenko's apparatus, the floridin being kept at 45—50°. The amylene residue in the floridin had a high refraction and sp. gr. and a reduced bromine number, showing that considerable change had taken place. The degree of unsaturation had decreased by 5·5%. Similar differences marked the behaviour of *iso*butylene with norit and floridin respectively. H. MOORE.

Oxidation of lubricating oil. H. MOORE and J. BARRETT (J. Inst. Pet. Tech., 1926, **12**, 582—585).—The property of oiliness does not come into action in machinery in which bearing surfaces are of adequate dimensions and bearing pressures moderate. Most failures in lubrication are occasioned by oxidation changes in the oil interfering with its regular supply to bearings. Oxidation leads to the formation of viscous or solid substances, and of jelly-like emulsions with water, and the necessity for an oxidation-resisting lubricant is marked in cases where the oil is returned to a sump and again fed to the machine. Experiments were made on 8 oils of approximately similar viscosity, with various bases, the Michie sludge test and Air Board oxidation test being applied, and hard asphaltum content, and viscosity and coke value before and after blowing being taken as an indication of liability to oxidation. In this respect the samples were found to vary greatly. H. MOORE.

Oxidation of transformer oils. R. SCHLAEPFER (Z. angew. Chem., 1927, **40**, 50—51; cf. Stäger, B., 1926, 331).—The apparent contradiction between the author's results and those of Stäger on sludge formation during the oxidation of transformer oils by air is due to the use by the latter of insufficiently refined products. A highly refined paraffin-base oil gives only oil-soluble acids on oxidation. A. B. MANNING.

Control of oils in newly-filled transformers. M. TILGNER (Chem.-Ztg., 1927, **51**, 43).—The oil of newly filled transformers should be examined as to quality. Poor oils decompose rapidly with the formation of water, acids, peroxides, and sludge, which are troublesome and dangerous. Even good oil deteriorates if subjected to imperfect purification methods, or by catalysis. There should be no separation of dark sludge insoluble in benzine after heating for 70 hrs. at 120°. W. G. CAREY.

Surface tension and wetting power of asphalt [and tar]. F. J. NELLENSTEYN (Chem. Weekblad, 1927, **24**, 54—58).—The wetting power of liquids is discussed in relation to capillary phenomena, and it is shown that the assumptions usually made are inconsistent. A low surface tension enables a liquid to spread over a smooth surface, but does not necessarily favour its rise in a capillary tube. The surface tensions of various mineral oil asphalts and of coal tars were determined at 180—200° by noting the differential rises in a wide and in a narrow capillary tube in an atmosphere of nitrogen; they vary between 23 and 25 dynes/cm.^{2} for the mineral asphalts; for coal tar the values found were 43·0, 40·9, and 32 at 20°, 50°, and 190° respectively. S. I. LEVY.

Gas-analysing apparatus. MCCOLLISTER and WAGNER.—See I.

Blast-furnace process. UHLMANN.—See X.

Uses of coke-oven gas in the steel industry. WILSON.—See X.

Aluminium as constructional material in industry. BUSCHLINGER.—See X.

PATENTS.

Manufacture of briquettes from lignite. C. L. MCCREA (U.S.P. 1,607,012, 16.11.26. Appl., 22.10.24).—A plant for briquetting raw lignite is combined with a lignite carbonising machine, the products from which pass through a steam-heated chamber in which the heavy tar constituents condense. The hot pitch and associated substances so produced are pumped in regulated amount to the stream of powdered lignite to be briquetted, as it is being fed to a drying and mixing chamber. The mixture of pitch and lignite is dried by contact with the hot gases from the carbonising machine, after which it is cooled and pressed into briquettes. S. PEXTON.

Apparatus for washing and sorting coal. F. BASCOUR (E.P. 256,211, 19.7.26. Conv., 28.7.25).—Powdered coal is allowed to gravitate down an inert column of water. Some distance down the column a stream of water is forced transversely across it through openings at substantially the same level. Valves and slides are provided whereby the transverse flow can be regulated in quantity and in its angular direction, so that the dense particles fall through the stream and the lighter particles pass away with the stream and are separately collected. S. PEXTON.

Dehydration of coal sludge. K. and K. WOLF (E.P. 260,002, 18.10.26. Conv., 17.10.25).—Coal sludges obtained from flotation or similar processes are mixed with dry coal or coke of 0—2 mm. size until the final product is capable of being handled with shovels or moulded into briquettes, the product having a water content of less than 25%. The mixture is dried by

exposure to air; the drying may be accelerated by using preheated air or drying drums. A coal sludge, containing 58·2% of water, when spread out in a layer 5 cm. deep, after 72 hrs.' exposure to air at 18° contained 47·5% of water; the same sludge mixed with coal and coke as described after 72 hrs.' drying contained 5·7% of water, or if briquetted and dried 1·3% of water.

A. C. Monkhouse.

Fuel-drying apparatus. H. Kreisinger, J. E. Bell, and J. Anderson, Assrs. to Combustion Engineering Corp. (U.S.P. 1,608,699, 30.11.26. Appl., 28.8.23).—In each chute supplying fuel to the pulverisers is provided a number of flue passages through which hot waste furnace gases are exhausted. S. Pexton.

Manufacture of agglomerated adsorbent carbon. Soc. de Récherches et d'Exploitations Pétrolifères (E.P. 244,461, 8.12.25. Conv., 9.12.24).—The adsorbent carbon is finely ground and mixed with an organic binder soluble in water, and dissolved in a solution of a soluble salt. The paste obtained is moulded to the shape required, dried in an oven, and then coated with an aqueous solution of an organic substance to which is added a soluble salt. The granules are then carbonised in a closed vessel, the coating forming an envelope to protect the granules from mechanical action. After washing with acid and water, a further heating at red heat is given in order to restore the adsorbent properties.

A. C. Monkhouse.

Production of active carbon in a pulverulent form at low temperatures. K. Bube (E.P. 262,278, 22.12.25).—Oils such as producer-gas tar or crude oil rich in asphalt are heated in a pressure-resistant apparatus at 350—500° in such a way as to avoid vaporising the whole of the oil. The carbon formed by cracking is protected from overheating by the presence of the liquid oil. The products are all discharged from a common outlet, and the pulverulent active carbon is purified by treatment with hot water. S. Pexton.

Hydrogenating and cracking carbonaceous matter. F. Bergius (U.S.P. 1,607,939, 23.11.26. Appl., 5.4.24. Conv., 31.5.23).—Hydrocarbons or carbonaceous materials are treated in a reaction vessel fed with hydrogen. The mixed gases and vapours are passed from the reaction sphere through a condenser followed by an oil washer in which the permanent gases are scrubbed under high pressure. The residual gases containing hydrogen are returned to the reaction vessel. The wash oil is stripped of its hydrocarbons, which are decomposed at a high temperature into hydrogen and carbon, the hydrogen being returned to the reaction chamber. S. Pexton.

Retort furnaces [for carbonisation of coal]. W. M. Carr, H. J. Toogood, and R. Dempster & Sons, Ltd. (E.P. 263,269, 6.11.25).—The furnace is heated by coal gas from the retort, and the combustion gases are utilised in a waste-heat boiler. To reduce the calorific value of the coal gas sufficiently to avoid deposition of lampblack on the walls of the retort, a portion of the combustion gases is mixed with it by utilising the pressure at the outlet of the boiler fan.

H. Holmes.

Annular hearth oven. E. W. Hartman, Assr. to Hartman Interests, Inc. (U.S.P. 1,608,597, 30.11.26. Appl., 20.11.24).—A cylindrical retort is provided centrally with a number of superimposed steam-tight chests. The flange plates, divisional between the successive chests, extend outwards from the chests, forming decks which retard the flow of material to be distilled in its downward passage through the retort. A mechanical agitator displaces the material from the successive decks. Independently regulated steam supplies are connected to each of the steam chests which form the heating elements. S. Pexton.

Ovens for distilling coal. J. Daniels (E.P. 239,884, 14.9.25. Conv., 13.9.24).—In a battery of retorts or ovens which has vertical combustion flues there is provided a series of roof-flues running lengthwise throughout the whole of the battery. These flues, besides collecting and transferring the combustion products from the up-flow flues to the down-flow flues, form channels for equalising the distribution of hot gases in the down-flow flues, and make for even temperatures not only in each oven, but throughout the battery. S. Pexton.

Treatment of peat or marsh soil to render them proof against moisture and fire. L. G. Wilkening (E.P. 244,716, 2.10.25. Conv., 17.12.24).—Peat, marsh soil, or substances containing vegetable mould are impregnated with a dilute solution of a halogen salt of an alkali metal or of ammonium. The excess solution is squeezed out, and the product when pressed forms material suitable for building or for insulating purposes.

S. Pexton.

Method and apparatus for the disposal of exhaust gases. A. E. White. From C. E. Thompson (E.P. 262,181, 3.9.25).—Exhaust gases from an internal-combustion engine are rendered innocuous and odourless by mixing with air, igniting electrically, and passing over a catalyst to promote combustion. In cases where the exhaust gases contain insufficient combustibles for the combustion to be self-supporting in the presence of the catalyst, means are provided for introducing fuel gas from the engine inlet to the exhaust prior to the treatment.

S. Pexton.

Apparatus for absorption of hydrocarbons. D. L. Newton (U.S.P. 1,608,416, 23.11.26. Appl., 19.7.24).—Gas to be scrubbed with a liquid medium is passed up a tower containing a number of trays down which the scrubbing medium flows. Each tray is sealed in the washing fluid to the height of an overflow through which liquid passes to the next tray below. The trays are perforated to allow for the passage of gas, each perforation being surmounted by a circulating tube having an open top and provided with side openings near the bottom. The circulating tubes are completely submerged in the washing medium, and are separated into rows by baffle plates. S. Pexton.

Generation of gaseous fuel. F. Umpleby (E.P. 263,561, 8.10.25).—A vertical retort is enclosed in a furnace consisting of mild steel plates lined with heat-insulating bricks. Colloidal, liquid, or pulverised fuel is admitted to the furnace through a series of nozzles pointing in a downward direction; the products of combustion are withdrawn at the top of the furnace and

pass through a waste-heat boiler. The ash is cooled by a series of water pipes at the bottom of the furnace, and removed automatically. Colloidal, liquid, or pulverised fuel is conveyed by means of an air or gas stream to the retort through an annular discharge pipe, and passes down through the distillation zone, which contains a vertical pipe for withdrawal of the gases, to the gasification zone, to which air and steam are admitted. The ash is ejected from the bottom of the retort by means of compressed air or steam, or if coke be formed this may be fed back to the furnace. A. C. MONKHOUSE.

Joint manufacture of carburetted water-gas and volatile hydrocarbon motor fuels. F. A. HOWARD, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,609,023, 30.11.26. Appl., 24.3.21).—In the manufacture of carburetted water-gas the oil feed is controlled with respect to the temperature of cracking, so that the resulting fixed hydrocarbons are from 15% to 60% by weight of the oil supplied to the carburettor. The mixed gases and vapours are cooled whereby the condensed oil containing the volatile hydrocarbon motor fuel is separated from the fixed gases, the calorific value of these being 450—650 B.Th.U./cub. ft. according to the rate of oil feed. S. PEXTON.

Apparatus for the destructive distillation of bones and the like. C. H. SHEARMAN (E.P. 263,659, 17.3.26).—A vertical steel cylinder lined with refractory material has a grate at the bottom supporting at its centre a short tube of firebrick perforated with holes. A layer of wood is placed on the grate and the cylinder filled with bones. As the charge burns, air vents, arranged in a staggered manner round the cylinder, are opened by the removal of firebricks. Low-pressure steam is admitted and the products of distillation are removed by a fan and pass down a chamber containing metallic pipes with insulated rods inside. These are connected to a high-tension current, and the liquid products condensed are removed at the base of the chamber.
A. C. MONKHOUSE.

Apparatus for continuous distillation [of oil]. F. M. HESS (U.S.P. 1,608,741, 30.11.26. Appl., 29.4.22). —A continuous still is built up in sections with heating plates inclined alternately to the right and to the left. Crude oil flows by gravity over the heating plates, and in its passage from one section to another passes through a seal which prevents the counter flow of the vapours. The vapours are withdrawn from each section and condensed in separate units, from which the several condensates are withdrawn. S. PEXTON.

Distillation of solid carbonaceous materials. J. F. RICHARDSON (U.S.P. 1,609,128, 30.11.26. Appl., 24.4.22).—Solid carbonaceous material is distilled in a combination vertical retort, the heat being supplied by the maintenance of a zone of combustion within the retort with a regulated supply of air. The air is admitted to the retort through a fluid-sealed inlet, the extent of the seal varying directly with the pressure in the retort in such a way that the rapidity of heating is controlled automatically by variations in the rate of air supply.
S. PEXTON.

Destructive decomposition of organic substances. J. H. WALLIN (U.S.P. 1,608,075, 23.11.26. Appl., 18.4.23. Conv., 26.8.20).—Organic substances are heated at not less than 250° and under 40—500 atm. pressure with sufficient caustic alkali to combine with all acids formed. S. PEXTON.

Pressure distillation of hydrocarbons. F. M. HESS (U.S.P. 1,610,523, 14.12.26. Appl., 7.6.22).—The distillation stock is heated under pressure by a fluid heating medium and hot residuum, each of which circulates in separate pipe systems within the still; the vapours and residuum pass, out of contact with each other, to a dephlegmator, reflux from the condensation of the vapours returns to the still, and uncondensed vapours pass to a condenser. W. N. HOYTE.

Cracking of [hydrocarbon] oils. U. S. JENKINS (E.P. 262,666, 25.6.26).—The cracking stock is fed to a still at a greater rate than is ordinarily required for cracking, and adsorptive material such as fuller's earth or lime is added. The oil is subjected in the still to heat and pressure, the vapours formed by cracking are conducted away, the residuum together with adsorptive material is continuously drawn off into an evaporator, and the pressure thereon is reduced to atmospheric. Further disengagement of vapours takes place, aided by live steam if desired. The residuum finally flows very slowly through a settling tank in which the adsorptive material together with suspended carbon are deposited. By the flushing action of the excess of oil and by the presence of the adsorptive material deposition of carbon in the cracking still is prevented. W. N. HOYTE.

Desulphurisation and hydrogenation of unsaturated and waste [hydrocarbon] oil. J. A. BISHOP (U.S.P. 1,610,242, 14.12.26. Appl., 9.4.23). —The oil is maintained at 450—500°, carbon monoxide and steam are introduced independently, carbon dioxide and nascent hydrogen are produced, and hydrogenation and the formation of hydrogen sulphide take place.
W. N. HOYTE.

Recovering resinous substances from cracked petroleum distillates. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,608,135, 23.11.26. Appl., 27.4.23).—Cracked petroleum distillates are distilled with, or filtered through, an adsorbing agent, such as bentonite, charcoal, or the like, and the adsorbent is extracted with a solvent, such as ether–alcohol, or liquid sulphur dioxide. T. S. WHEELER.

Method and apparatus for determining, indicating, and recording the calorific value of liquid and gaseous fuels. J. M. MOREHEAD (U.S.P. 1,607,684, 23.11.26. Appl., 2.2.23).—Liquid or gaseous fuel is burned at a measured rate in a still which has the form of a vertical gas-tube boiler, the tubes forming flues whereby the products of combustion are conveyed to a condensing coil immersed in a vessel through which liquid flows into the calorimeter around the tubes. The liquid in the calorimeter boils and distils into a second condensing coil which drains into the same outlet as the first, both coils being in the same condensing bath. The rate at which the condensate collects is recorded, or alternatively can be measured over a suitable time interval. S. PEXTON.

Fuel for internal combustion engines. E. G. E. MEYER (E.P. 262,363, 12.6.25).—Oil of relatively high b.p.

is mixed thoroughly with 4% by vol. of ethyl ether and 1 pt. by wt. in 10,000 of ammonia solution; if required 10% by vol. of volatile hydrocarbon is added. A satisfactory motor fuel is produced. The addition of ammonia causes the retention of the ether in the mixture, and prevents gum formation on the valves.

W. N. Hoyte.

Tool for repairing gas retorts, furnaces, etc. R. J. Milbourne and C. Poulson (E.P. 263,419, 29.7.26).

Atmospheric gas burners. Cannon Iron Founders, Ltd., and H. S. Hawthorne (E.P. 263,335, 11.2.26).

Apparatus for storing inflammable liquids or for supplying such liquids. E. G. E. Beaumont, and Anglo-American Oil Co. (E.P. 262,613, 8.1.26).

Feeding solids into or removing solids from vessels under pressure [*e.g.*, for hydrogenation of coal] (E.P. 262,901).—See I.

Dispersing substances in water (U.S.P. 1,609,308).—See I.

III.—TAR AND TAR PRODUCTS.

Main constituents and possible utilisation of primary tar. C. J. Ward (J. Inst. Pet. Tech., 1926, **12**, 591—597).—The author reviews researches which have been made on primary (low-temperature) tars. A fresh tar produced from a blend of Arley and Butterley coals, carbonised at 620°, was examined. Solids were precipitated by addition of 400 c.c. of ether to 20 c.c. of tar. Treatment with sodium carbonate solution, dilute sulphuric acid, and caustic soda solution, and further extraction with ether resulted in a yield of 3·8% of solids, 27·09% of phenols, 6·63% of bases, and 59·11% of neutral oil of *d* 0·975. Similar treatment of a tar which had been kept several months gave 12·66% of solids. Continuous precipitation of solids indicates this to be a time reaction.

H. Moore.

Sorption of the vapours of benzene and toluene by silicic acid gel impregnated with carbon (carbo-gel). H. A. Fells and J. B. Firth (J.S.C.I., 1927, **46**, 39—40 T).—Silica gels uniformly impregnated with carbon ("carbo-gels") have been prepared, and their sorptive capacities determined for benzene and toluene vapours over the range pure gel to pure carbon. The results show that the sorptive capacity of the gel increases with rise of carbon content to a maximum at about 18% of carbon, and then continuously decreases.

Valuation of pitch as a binding material for carbon electrodes. G. Schuchardt (Chem.-Ztg., 1927, **51**, 42).—The usual tests are of little value for determining the suitability of pitch for carbon electrodes. For the determination of carbon 0·5 g. of the pitch is boiled for 1 hr. with 50 c.c. of water-free tetralin under a reflux condenser, and, after cooling, the mixture is filtered through a Gooch funnel and the residue is washed with warm tetralin, dried, and weighed. The coking test is most suitable for judging the pitch, and may be made by heating a small block of the electrode mixture in an iron tube closed at one end for 15—20 min. in a forge stove. Preferably, however, the test is made by coking a whole electrode; this takes several hours, but information as to the quality of the electrode itself is also obtained. Pitch for electrodes should have a low ash, a softening point of about 75°, and should coke quickly, giving a high yield of hard coke.

W. G. Carey.

Surface tension and wetting power of asphalt [and tar]. Nellensteyn.—See II.

Patents.

Production of bitumen emulsion. C. H. Thompson and W. J. McGivern (E.P. 263,307, 5.1.26).—Stable emulsions suitable for road dressing are prepared by adding 3—7 pts. of "liquid rosin" and then ½—1 pt. of solid caustic soda in 15 pts. of water to 50 pts. of melted bitumen, the temperature being kept constant and the mass vigorously agitated. "Liquid rosin," which contains a large proportion of resinic acids, is a by-product in the treatment of wood with sulphite liquor.

S. Pexton.

Manufacture of phenolic compounds. W. J. Hale and E. C. Britton, Assrs. to Dow Chemical Co. (U.S.P. 1,607,618, 23.11.26. Appl., 7.1.24. Cf. U.S.P. 1,213,142—3; B., 1917, 382).—Chlorobenzene is heated with 1 or 2 mols. of sodium hydroxide and water at 380° for 1 hr. in presence of 10% by weight of diphenyl oxide, which depresses the formation of further quantities of itself, so that phenol is obtained in satisfactory yield.

T. S. Wheeler.

Production of active carbon (E.P. 262,278).—See II.

Composition of bitumen and rubber (E.P. 263,028).—See XIV.

IV.—DYESTUFFS AND INTERMEDIATES.

Patents.

Manufacture of yellow azo dyestuffs. I. G. Farbenind. A.-G., Assees. of A. Dorrer (U.S.P. 1,610,936, 14.12.26. Appl., 20.11.25. Conv., 15.12.24).—Monoazo dyestuffs, obtained by coupling a diazotised naphthylaminemonosulphonic acid with diphenylamine, are treated, preferably after conversion into the corresponding nitroso-derivatives, with a mixture of nitric and sulphuric acids, so that mono- and di-nitro-derivatives are obtained. The products dye silk in a bath containing soap and sodium sulphate, and also wool, paper, and leather.

T. S. Wheeler.

Direct blue disazo dyestuff. P. Caccia (U.S.P. 1,609,793, 7.12.26. Appl., 25.9.25).—1 : 8-Aminonaphthol-3 : 6-disulphonic acid (1 mol.) and 2-naphthylamine-5-sulphonic acid (1 mol.) are boiled with sodium cyanide solution (0·2%), mixed, and poured into diazotised benzidine (1 mol.). The *product*, which is separated by addition of sodium carbonate and sodium chloride, is probably the disazo dye with the two amino-groups converted into cyanamido-groups. It dyes artificial silk light blue shades in a bath containing soap and sodium carbonate, and navy blue shades in a sodium sulphate bath.

T. S. Wheeler.

Trisazo dye. Tetrakisazo dye. J. B. Oesch, Assr. to Newport Co. (U.S.P. 1,605,973—4, 9.11.26. Appl., [A, B], 19.9.25).—(A) Diazotised acetamido-Cleve's acid (4-acetamido-1-naphthylamine-6- and -7-sulphonic acids)

or (B) a diazotised aminoazo-dyestuff, such as 4-amino-2 : 2′-dimethylazobenzene-6 : 4′-disulphonic acid, is coupled with *m*-toluidine, the product is diazotised and coupled with *m*-toluidine, and this product is diazotised and coupled with salicylic acid, or an alkyl or halogen derivative, to yield *substances* which dye unmordanted cotton orange to brown shades, very fast to light.

T. S. Wheeler.

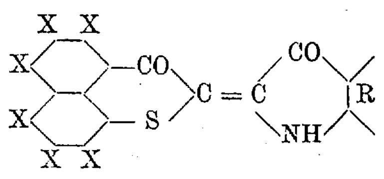
Manufacture of 1 : 4-diarylamino-5 : 8-di[hydr]-oxyanthraquinones. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 263,370, 13.4.26).—The condensation of leuco-1 : 4 : 5 : 8-tetrahydroxyanthraquinone with arylamines is carried out in presence of arsenic acid as oxidising agent. *E.g.*, 4 pts. of leuco-1 : 4 : 5 : 8-tetrahydroxyanthraquinone are dissolved in 40 pts. of *p*-toluidine, and 1 pt. of arsenic acid is added at about 100°. After 15 min., 0·8 pt. of boric acid is added. The reaction begins at once, and is complete in 1—1½ hrs. The product is worked up in the usual way.

A. Davidson.

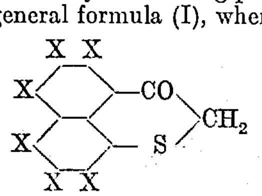
Vat dyestuffs of the anthraquinone series. I. G. Farbenind. A.-G., Assees. of F. Funcke (U.S.P. 1,609,084, 30.11.26. Appl., 17.9.25. Conv., 27.9.24).—Aryl-3 : 3-dichloro-2-keto-1 : 2-dihydro-*p*-thiazines are condensed with *o*-arylenediamines to give, if one of the components is an anthraquinone derivative, products which dye cotton yellow to brown in a hyposulphite vat. The shades are fast to alkali if an alkylated or arylated arylenediamine is used, but if not, they can be rendered so by alkylation or arylation, *e.g.*, by treatment with ethyl *p*-toluenesulphonate. The condensation is carried out by heating the compounds in a high-boiling solvent in presence of a condensing agent. *E.g.*, αβ-anthraquinone-3 : 3-dichloro-2-keto-1 : 2-dihydro-*p*-thiazine (7·5 pts.) is heated with 1 : 2-diaminoanthraquinone (4·8 pts.) in nitrobenzene (80 pts.) containing anhydrous sodium acetate (3·5 pts.) for 2—3 hrs. at 145° to give a *product* which on ethylation dyes cotton fast yellow shades.

T. S. Wheeler.

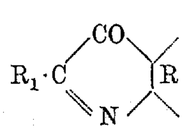
Vat dyestuffs of the 2-thionaphthen-2-indoleindigo series. R. Herz and J. Müller, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,613,275, 4.1.27. Appl., 23.3.26. Conv., 25.10.24).—Vat dyes of the probable general formula :—



are obtained by condensing perinaphthoxypenthiophens of the general formula (I), where X = H, or a univalent



(I)



(II)

substituent, with α-isatin derivatives of the general formula (II), where R is a benzene or naphthalene residue, possibly substituted, and R_1 a halogen or an arylido-group. The *dyes* produced dye cotton and wool fast blue to green shades.

A. Davidson.

Manufacture of sulphur dyestuffs. I. G. Farbenind. A.-G., Assees. of J. Wukte and W. Hagge (U.S.P. 1,609,927, 7.12.26. Appl., 8.6.25. Conv., 14.6.24).—In the manufacture of sulphur dyes as described in F.P. 343,377, E.P. 14,543/05, G.P. 179,021 (B., 1904, 1026; 1906, 260; 1907, 681), in which an organic compound is treated with sulphur in presence of metallic copper or a copper salt, the formation of copper sulphide is inhibited, and a dye giving clearer shades is obtained if the copper is added in the form of potassium cuprocyanide, or if the copper salt usually employed, *e.g.*, copper sulphate, is mixed with an alkali cyanide.

T. S. Wheeler.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Conversion of fibroin, chitin, casein, and similar substances into the ropy-plastic state and colloidal solution. P. P. von Weimarn (Ind. Eng. Chem., 1927, **19**, 109—110).—Fibroin in the form of silk wadding possesses higher colloidal dispersability in aqueous salt solutions than cellulose, and 30% colloidal solutions in such salts as calcium chloride and calcium nitrate are possible. A filiform coagulum can be prepared by addition of ethyl alcohol to a 10% solution in calcium nitrate cooled from 115° to 50—25°. Colloidal solutions of chitin are very similar to those of cellulose; casein is readily dispersed in lithium thiocyanate solutions, whilst fibrin and keratin require extremely soluble salts in highly concentrated solution. Rate of dispersion depends not only upon the salt used and its concentration and temperature, but also upon the life history, age, previous treatment, and purity of the preparations used. The same range of inequalities appears to hold for all the above substances, thus:

$$LiCNS > LiI > LiBr > LiCl;\ NaCNS > NaI;$$
$$Ca(CNS)_2 > CaI_2 > CaBr_2 > CaCl_2.$$

D. G. Hewer.

Determination of copper in specially treated fabrics. L. Levi (Ann. Chim. analyt., 1927, [ii], **9**, 2—7).—Hemp, linen, and cotton materials subject to the action of fermenting media are protected by treatment with insoluble metallic soaps (chiefly copper) with or without previous treatment with aniline black and fixation with potassium dichromate. Determinations of copper show that when the raw material has been directly treated with a copper soap, all the copper passes into a nitric acid solution of the calcined product, but with the aniline treatment a product of the form $CuCr_2O_4,2CuO$ is formed which on calcination gives the soluble copper oxide and insoluble $CuCr_2O_4$. In order to determine the total copper 1 dm.^{2} of material is calcined, the ash treated with boiling 50% nitric acid, filtered, any soluble copper washed out of the insoluble residue, which is then calcined in a platinum crucible and the residue treated with 3 g. of Meker's oxidising mixture. After cooling, the ash is dissolved in dilute nitric acid, the solution added to the first, and the mixture electrolysed. To determine copper insoluble in water 1 dm.^{2} of material is washed for 12 hrs. in running water and treated as above.

D. G. Hewer.

Nitration of cellulose in the presence of phosphoric acid. C. K. Krauz and F. J. Blechta (Chem. News, 1927, **134**, 1—4, 17—20).—The substitution of phosphoric acid for sulphuric acid in the mixed acid used for the nitration of cellulose would eliminate the costly boiling and pulping processes at present necessary for the removal of sulphuric esters, which are the chief cause of instability in cellulose nitrate made by the usual method. The product is sufficiently stable to be used in explosives manufacture after washing with cold water until neutral, owing to the non-formation of phosphoric esters of cellulose during nitration. The action of an anhydrous mixture of nitric and phosphoric acids (1 : 1) leads to a cellulose nitrate containing 13·0% N, which is the most highly nitrated product obtainable with these two acids. The addition of water to this mixture causes a rapid fall in nitrogen content, and with acids containing more than 3·5% of water gelatinisation occurs. An acid containing 18% of water produces a cellulose nitrate having only 4·9% N, whereas a nitric–sulphuric mixture of corresponding composition gives a product containing 12% N. The optimal proportion of nitric to sulphuric acid in mixed acid is usually 1 : 3, but that of nitric to phosphoric acid is 1 : 1. As the proportion of phosphoric acid is increased beyond this ratio, the nitrogen content of the product falls continuously. The addition of 5% of sulphuric acid to the nitric–phosphoric acid mixture causes the formation of cellulose sulphuric esters. Nitration takes place much more slowly with nitric–phosphoric acid than with nitric–sulphuric acid, 4 hrs. being required for complete nitration in the case of the former mixture. Below 30° the nitration proceeds normally, but at higher temperatures products of lower nitrogen content and higher solubility in waste acid are formed. The rôle of the phosphoric acid in the nitric–phosphoric acid mixture appears to be that of a diluent.

W. J. Powell.

Resistance of cellulose to heat and its absorptive power for gases. D. Costa (Annali Chim. Appl., 1926, **16**, 636—647; cf. A., 1925, ii, 956).—Experiments made with cotton wool and with filter-paper cellulose subjected to various heat treatments show that the absorptive power of cellulose for ammonia, hydrogen chloride, sulphur dioxide, and carbon dioxide bears no relation to the length or physical structure of the fibre, but varies appreciably with the chemical character of the complex. When heated in a bath of inert material, the cellulose fibre resists the prolonged action of temperatures up to 230° without substantial change in either morphological or absorptive properties. T. H. Pope.

Resistance of cellulose to heat. II. D. Costa (Annali Chim. Appl., 1926, **16**, 647—652; cf. preceding abstract).—When heated in a paraffin bath, cellulose begins to decompose at 260—270°, emitting gases and volatile products consisting principally of carbon dioxide and water which are formed at the expense of the oxygen of the cellulose molecule itself. As the temperature is raised the decomposition progresses, until at 365° the fixed residue has the composition C 83·11%, H 6·4%, O 10·49%, which corresponds approximately with the mean composition of gas coal, namely, C 80—85%, H 5·8—5·0%, O 14·2—10·0% (cf. Bergius, B., 1912, 761). After precipitation by means of Schweizer's reagent, cellulose exhibits less resistance towards heat than the original fibre, but the residue obtained at 365° is of similar composition to that given above. T. H. Pope.

Fluorescence of wood cellulose and vegetable tanning extracts. O. Gerngross [with N. Bán, G. Sándor, and K. Tsou] (Papier-Fabr., 1927, **25**, 49—52).—The admixture of synthetic tanning agents or substitutes derived from sulphite liquor with natural tanning extracts can be detected by examining the colour and intensity of the fluorescence of the diluted extract in ultra-violet light. Most of the synthetic products exhibit a characteristic violet fluorescence which, in the case of extracts derived from sulphite liquor, changes to green on making the solution alkaline. Cellulose fibres when dipped in tanning solutions irreversibly adsorb some of the fluorescent substance; thus cotton, wool, viscose silk, hydrocellulose, and starch after immersion in quebracho or tizerah extracts appears yellow (B., 1925, 643), while pine or larch bark extracts cause them to exhibit a violet fluorescence. Diacetylcellulose and acetate silks readily adsorb both the violet and yellow fluorescent material. Nitrocellulose does not adsorb the violet substance, and the amount of the yellow substance adsorbed depends on the extent to which the cellulose is nitrated. A nitrocellulose containing 11% N exhibits a strong yellow fluorescence after immersion in quebracho extract, but a guncotton of 13·2% N has very little adsorptive action. Sucrose, lactose, dextrose, cystine, tyrosine, and glycine anhydride exhibit no fluorescence with either solution. The violet fluorescence observed in sulphite liquor, when exposed to ultra-violet light, is also shown by unbleached sulphite-cellulose (B., 1926, 911). Lignin, on treatment with calcium bisulphite solution, also yields a solution having a violet fluorescence, but cotton wool dipped in this solution remains practically colourless. Solution-fluorescence and fibre-fluorescence are therefore due to the presence of different substances. Wood chips when heated with water at 115° give a liquor which causes fibre-fluorescence. It is, therefore, unlikely that this property is due to the presence of lignosulphonic acid, but to that of some substance already in the wood which has similar properties to, and may be identical with, the fluorescent principle of pine bark extract. It is sensitive to alkalis and bleaching agents, hence bleached and soda cellulose do not exhibit fluorescence. W. J. Powell.

Preparation of sugar from sawdust. E. Hägglund (Papier-Fabr., 1927, **25**, 52—60).—Highly concentrated hydrochloric acid has, in comparison with sulphuric acid, a low solvent power for cellulose. With the latter one part of acid to one of cellulose suffices for complete solution, but with hydrochloric acid the proportion necessary is 7 : 1. The use of such quantities of acid commercially is uneconomical, but since an equilibrium is set up between the acid solution and the carbohydrates in the wood, and a sugar–hydrochloric acid solution which has completed its reaction with one charge of sawdust still has the power to hydrolyse part of the cellulose in a fresh charge, the difficulty has been overcome by the use of a continuous process (B., 1924, 593). A 30% sugar solution containing about 30% of hydrochloric acid and practically carbohydrate-free lignin are the

products obtained. The concentration of sugar can be raised to 44% by working at 30—40°, but with highly concentrated sugar solutions reversion to *iso*maltose takes place. The hydrochloric acid is removed by distillation under reduced pressure, the heat being supplied by mixing hot oil with the acid sugar solution. The resultant syrup (sugar 56%, hydrochloric acid, 9%) is dried by a current of hot air, the final product containing only 1% of hydrochloric acid. This is suitable without further purification for the manufacture of alcohol. Sugar obtained from fir wood consists of dextrose 61·9%, mannose 24·7%, galactose 4%, lævulose 1·4%, and xylose 8%. W. J. POWELL.

Relation between analytical "chlorine values" and factory bleach consumptions for wood pulps. G. K. BERGMAN (Papier-Fabr., 1926, **24**, 742—746).—Careful observations were made on the bleaching of various types of wood pulp on the large scale according to standard procedure. The pulp at about 4% concentration was treated with a known quantity of bleach liquor slightly in excess of the expected consumption, the temperature being raised during 1½ hrs. to 40°, the total time of bleaching being 5 hrs. Tests were made every ½ hr. of the fall in alkalinity and the rate of disappearance of active chlorine. A correction, equivalent to ¼ hr. further bleaching, was added at the end to compensate the continued action during washing off. Typical values for actual consumptions were :—Sulphite pulps, easy bleaching, different cooks, 3·17, 4·41, 4·36, 3·09; sulphate pulp, easy bleaching, 5·05; aspen sulphite, 2·55, all values being in terms of active chlorine consumed per cent. of dry cellulose. In practice, an excess over the actual consumption is always used to stimulate the operation, such excess being a matter of judgment and economics. Comparing the mill results with the indications given by various laboratory methods, it was found that the Bergman method (B., 1923, 9 A) gave the closest concordance, maximum error ± 10%. The Tingle chlorine factor (B., 1922, 137 A) gave too high results for the sulphite and too low for the sulphate pulps. A modification of the standard German "chlorine value" method, devised by Enso for factory control and commonly used in Finland, gave results generally too low; it is, however, possible to determine an expression for converting Enso values into Bergman values for sulphite and sulphate pulps respectively.

J. F. BRIGGS.

PATENTS.

Method of reeling-off silk cocoons. B. LOEWE (U.S.P. 1,607,959, 23.11.26. Appl., 27.12.23. Conv., 24.1.23).—The cocoons are treated, without previous degumming, in a cold aqueous solution of at least one substance containing an acid radical until the silk-worm pupæ are killed. The cocoons are then immersed for not less than a minute at 25° or less in an alkaline bath, and reeled off at the ordinary temperature without using hot water. The silk may be subsequently degummed in hot water without the addition of soap.

D. J. NORMAN.

Yarn dryer. H. HAAS (E.P. 263,463, 12.10.26).—Hanks of light material such as silk or artificial silk are submitted to an ascending stream of hot air in a preliminary chamber of a drying tunnel, the lower portions of the hanks being thereby heated to a temperature higher than that of the succeeding chambers, whilst in the latter complete drying without tangling is obtained by means of descending currents of hot air.

B. P. RIDGE.

Production of cellulose pulp. A/S. RAOUL PICTET, & F. THARALDSEN (E.P. 252,344, 26.4.26. Conv., 20.5.25).—Wood or like material is digested with aqueous solutions containing more than 100 g./litre of sulphur dioxide at such temperature, preferably below 110°, that a pressure in excess of 8 atm. is developed in the digester. The process is complete in 8—16 hrs., and gives high yields of pulp of good colour. D. J. NORMAN.

Filtering wood pulp. J. B. BEVERIDGE (U.S.P. 1,603,507, 19.10.26. Appl., 16.1.26).—The alkali-laden foam produced during the filtration of wood pulp made by the alkaline process is broken down, and the alkali trapped, by passing the foam-laden air through a liquid bath on its way to the pump. D. J. NORMAN.

Manufacture of artificial silk threads from viscose. H. BASS, and ERSTE BÖHMISCHE KUNSTSEIDE-FABR. A.-G. (E.P. 260,681, 5.8.25).—A spinning bath for viscose silk contains, in addition to sodium bisulphate, an excess either of sodium sulphate or of sulphuric acid. An excess of sodium sulphate imparts to threads of medium and high denier excellent lustre and softness, whilst an excess of sulphuric acid is particularly useful when filaments of 2—3 deniers are required. The addition of both molasses or distiller's wash and water-soluble salts of multivalent metals favourably affects the softness, lustre, and covering power of the threads. A suitable spinning bath contains sodium sulphate (22 pts.), magnesium sulphate (15 pts.), sulphuric acid (11 pts.), molasses (15 pts.), and water (37 pts.). D. J. NORMAN.

Process and apparatus for producing artificial silk filaments. K. GRUNERT (E.P. 263,462, 8.10.26).—A cellulose solution is squirted through an open-topped vertical funnel containing a descending column of precipitating liquid, the velocity of which is regulated by the flow of an opposing column meeting the first near the outlet of the funnel, and the filaments formed are drawn off in the form of a spun thread. The apparatus comprises a number of squirting tubes arranged above corresponding outlets in the floor of a tank, the height of liquid in the tank determining the velocity of outflow from the tubes. B. P. RIDGE.

Production of dialysing and pervaporating membranes. P. A. KOBER (U.S.P. 1,608,886, 30.11.26. Appl., 17.6.18).—The membranes are formed from a solution containing nitrocellulose and a peptone.

D. J. NORMAN.

Manufacture of nitrocellulose film. J. A. WILSON, Assr. to DURATEX CORP. (U.S.P. 1,603,499, 19.10.26. Appl., 22.10.20).—Newly-made celluloid is coated with a film-forming solution containing nitrocellulose, a non-drying vegetable oil, a solvent, and a diluent in order to prevent loss of camphor. D. J. NORMAN.

Treating waste sodium monosulphite liquors. V. DREWSEN (U.S.P. 1,605,925—8, 9.11.26. Appl., [A, B, C], 3.4.22 and [D] 9.10.22).—(A) The liquor is concentrated, treated with caustic lime, incinerated, and

extracted with water to recover sodium carbonate. (B) The concentrated liquor is mixed with 20—30% (by weight) of limestone and burnt in, *e.g.*, a rotary furnace in the presence of air to form calcium sulphide and sodium carbonate. The latter is extracted from the pulverised residue with water, and converted into sodium sulphite by the action of sulphur dioxide, whilst the calcium sulphide is suspended in water and treated with carbon dioxide to liberate hydrogen sulphide for use in the preparation of sulphur dioxide. (C) The waste liquor is dried, incinerated, and extracted with water ; the resulting sodium sulphide is converted into sodium bicarbonate and the sulphur into sulphur dioxide. (D) A mixture of an excess of lime and nitre cake which has been causticised with powdered quicklime is incorporated with the concentrated liquor ; the mass is then dried and incinerated to form calcium sulphide and sodium carbonate, which latter is recovered by extraction with water. D. J. Norman.

Treating waste magnesium monosulphite cooking liquor. V. Drewsen, Assr. to West Virginia Pulp and Paper Co. (U.S.P. 1,605,929, 9.11.26. Appl., 17.12.24).—The liquor is boiled with slaked dolomitic lime to precipitate magnesium hydroxide. This is recovered and converted into bicarbonate by the action of a solution of carbon dioxide under pressure ; the resulting solution is filtered and treated with magnesium acid sulphite to regenerate magnesium sulphite.
D. J. Norman.

Treatment of black liquor. Treatment of residual liquor [from wood pulp manufacture]. L. Bradley and E. P. McKeefe, Assrs. to Bradley-McKeefe Corp. (U.S.P. 1,606,338 and 1,606,501, 9.11.26. Appl., [A] 19.5.21 ; [B] 3.5.26).—The residual black liquor from alkaline wood-pulp digesters is sprayed into a tower up which hot gases containing (A) carbon dioxide or (B) sulphur dioxide, or both, are passed. Under the action of these gases the liquor is concentrated, carbonated or sulphited, and organic matter is precipitated. The product is filtered, causticised to any desired extent, and re-employed in the digester.
T. S. Wheeler.

Manufacture of artificial threads, ribbons, films, and the like of viscose. A. Kämpf (U.S.P. 1,614,036, 11.1.27. Appl., 27.1.26. Conv., 6.8.21).—See E.P. 184,450 ; B., 1923, 711.

Production of acylated cellulose ethers. W. H. Glover and E. van Weyenbergh, Assrs. to Courtaulds, Ltd. (U.S.P. 1,613,451, 4.1.27. Appl., 22.7.25. Conv., 15.9.24).—See E.P. 241,679 ; B., 1926, 48.

Ageing apparatus for treating fabrics. J. Wood (E.P. 263,246, 7.10.25).

Artificial silk and like spinning machines. J. L. Rushton (E.P. 263,239, 26.9.25).

Hydro-extractors (E.P. 263,303).—See I.

Production of bitumen emulsion (E.P. 263,307).—See III.

Dyeing cellulose compounds [artificial silk] (U.S.P. 1,609,702).—See VI.

[Electrolyte for] electric batteries (E.P. 263,587).—See XI.

Making [aqueous] solutions of resins (E.P. 263,393).—See XIII.

VI.—BLEACHING ; DYEING ; PRINTING ; FINISHING.

Bleaching of wood pulps. Bergman.—See V.

Patents.

Dyeing cellulose compounds [artificial silk]. W. Duisberg, W. Hentrich, and L. Zeh, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,609,702, 7.12.26. Appl., 18.3.25. Conv., 18.3.24).—Cellulose esters and ethers can be dyed a variety of brilliant shades by treatment with sulphomethylaminoanthraquinone derivatives. *E.g.*, sodium 1 : 4-diaminoanthraquinone-*N*-methyl-ω-sulphonate (6 pts.), dissolved in water (10,000 pts.), gives bright clear violet shades on cellulose acetate silk at 70° in ¾ hr. T. S. Wheeler.

Process of dyeing and printing acetylcellulose. H. Hoz, W. Bernoulli, and A. Link, Assrs. to Soc. Anon. J. R. Geigy (U.S.P. 1,613,228, 4.1.27. Appl., 5.3.24. Conv., 31.3.23).—See E.P. 231,897 and 213,593 ; B., 1925, 588, 708.

Compositions for colouring hair. C. C. Huffman (E.P. 263,349, 6.3.26).

VII.—ACIDS ; ALKALIS ; SALTS ; NON-METALLIC ELEMENTS.

Volatility of the compounds formed by heating barium sulphate with sulphuric acid. F. Krauss (Chem.-Ztg., 1927, **51**, 38).—An acknowledgment of Hahn's criticism (B., 1927, 106) of the author's discovery that barium sulphate moistened with sulphuric acid imparts a green colour to the Bunsen flame. The origin of the colour is the subject of further investigations.
A. R. Powell.

Action of acetic acid on red lead. Formation of basic lead peracetate. C. Brückner (Chem.-Ztg., 1927, **51**, 55).—Glacial acetic acid reacts with red lead to form lead acetate and basic lead peracetate, the bulk of the latter crystallising out as a mass of microscopic white needles, addition of water to which results in hydrolysis with the formation of lead peroxide. Even with 40% acetic acid the momentary formation of basic lead peracetate may be observed ; this compound is therefore an intermediate product in the reaction, $Pb_3O_4 + 4Ac\cdot OH = 2Pb(OAc)_2 + PbO_2 + 2H_2O$.
A. R. Powell.

Plant for the production of carbon monoxide. —F. W. Burstall and S. J. Ellis (J.S.C.I., 1927, **46**, 35—36 T).—A short description of a plant for the production of carbon monoxide using sulphuric and formic acids in electrically heated reaction vessels.

Oxidation of ammonium sulphide. Applebey and Lanyon.—See II.

Lixiviation of copper minerals. Fester and Bertucci.—See X.

Selenium in rubber compounds. Boggs and Follansbee.—See XIV.

Direct titrimetric methods for magnesium, calcium, and sulphate ions, and their application in water analysis. Schoch.—See XXIII.

Patents.

Manufacture of chemically pure sulphuric acid. M. Krafft (U.S.P. 1,611,534, 21.12.26. Appl., 5.4.23. Conv., 10.4.22. Cf. E.P. 220,413; B., 1924, 868).—The acid content of gases from the lead chamber is removed by passing them through an acid-proof filter tower before they are returned to the chamber.

H. Royal-Dawson.

Manufacture of sodium bicarbonate and production of nitrogen. E. E. Arnold, Assr. to Nitrogen Corp. (U.S.P. 1,611,401, 21.12.26. Appl., 21.10.24).—Waste blast-furnace gases are burned in air, thus converting the carbon monoxide and hydrogen contents into carbon dioxide and water with a proportionate increase of nitrogen. The gaseous mixture is then compressed and passed through ammoniated brine.

H. Royal-Dawson.

Synthetic production of ammonia. C. Mott, Assr. to Compressed Gas Corp. (U.S.P. 1,611,359, 21.12.26. Appl., 9.10.22).—A mixture of nitrogen, oxygen, and an inert gas is passed over an oxidisable material to remove oxygen. The purified nitrogen is then mixed with hydrogen and passed through a device causing a proportion of the nitrogen to combine with the hydrogen. On removal of the ammonia produced, the residual gaseous mixture is passed through another synthesising device and the ammonia again removed, this process being continued till the percentage of inert gas is materially increased, the residue from the last separating device being used to reduce the material oxidised in the first part. H. Royal-Dawson.

Manufacture of aluminium chloride. R. J. Dearborn, Assr. to Texas Co. (U.S.P. 1,605,098, 2.11.26. Appl., 29.11.21).—A heated mixture of an aluminium ore, such as bauxite, and carbonaceous material is introduced into the top of a vertical retort, in which it is treated with chlorine or hydrogen choride at such a temperature that only the iron present is converted to chloride, which volatilises. The material then falls into a lower vertical retort maintained at a higher temperature, so that on treatment with chlorine or hydrogen chloride, aluminium chloride is formed.

T. S. Wheeler.

Production of calcium arsenate. E. A. Taylor, Assr. to Grasselli Chemical Co. (U.S.P. 1,612,233, 28.12.26. Appl., 14.4.25).—A mixture of lime and white arsenic is heated in the presence of an oxidising medium, the heat of the reaction being so conserved that only sufficient additional heat is supplied to complete the oxidation of the arsenite to arsenate.

H. Royal-Dawson.

[Manufacture of] sodium fluoride. W. Seigel (F.P. 607,546, 8.12.25).—Potassium silicofluoride is treated with sodium carbonate, hydrogen carbonate, or hydroxide, etc. in amount less than sufficient for the theoretical conversion: $K_2SiF_6 + 3Na_2CO_3 = 6NaF + K_2CO_3 + SiO_2 + 2CO_2$. A. Cousen.

Apparatus for the production of oxygen. Soc. Anon. le Salvoxyl (E.P. 252,318, 16.12.25. Conv., 23.5.25. Cf. E.P. 252,319; B., 1926, 744).—A portable apparatus for therapeutic uses for producing oxygen by the action of an anhydrous alkaline oxide on an excess of hydrogen peroxide. H. Royal-Dawson.

Production of chlorine and aluminium chloride. E. R. Wolcott, Assr. to Texas Co. (U.S.P. 1,612,686, 28.12.26. Appl., 28.2.21).—Hydrogen chloride is passed over heated alkaline-earth oxide, followed by an excess of oxygen in contact with the resulting metallic chloride, thus producing the oxide and free chlorine; the excess of oxygen contained in the chlorine mixture is reduced, and the reduced gas mixture is passed in contact with the compound of aluminium containing oxygen together with heated carbon to produce volatile aluminium chloride, which is separated from the resulting gases, the latter being treated to regenerate hydrochloric acid gas to be used again in the cyclic process.

H. Royal-Dawson.

Process for mining sulphur. W. T. Lundy and H. S. Burns, Assrs. to Freeport Sulphur Co. (U.S.P. 1,612,453, 28.12.26. Appl., 5.8.26).—A mixture of hot water and finely-divided earthy material, having a viscosity greater than that of the water, is injected into the sulphur-bearing formation. H. Royal-Dawson.

Synthesis of ammonia [dehydration of gases]. G. Claude, Assr. to Lazote Inc. (U.S.P. 1,609,058, 30.11.26. Appl., 19.7.21. Conv., 4.10.18).—See E.P. 129,637; B., 1920, 63 a.

Production of titanium oxide. P. A. Mackay, Assr. to National Metal and Chemical Bank, Ltd. (U.S.P. 1,613,234, 4.1.27. Appl., 15.5.26. Conv., 28.5.25).—See E.P. 256,734; B., 1926, 876.

Treating monosulphite liquors (U.S.P. 1,605,925—9).—See V.

Electrode for ozone production (E.P. 263,356).—See XI.

VIII.—GLASS; CERAMICS.

Ageing of thermometer glasses. L. Holborn and J. Otto (Z. Instrumentenk., 1926, **46**, 415—424; Chem. Zentr., 1926, II, 1782—1783).—Tests on various types of glass (Jena 59 III and 16 III, Supremax, and hard Thuringian) showed that, in order to age thermometer glass, it was necessary to heat the finished ware to some 20—30° above the initial temperature of the critical zone of the thermal expansion for 3 hrs., then to cool it in about the same time, and to hold at the softening limit for 6 hrs. The method of subsequent cooling had but little influence. At the highest temperature employed the glass was still so viscous that there was no appreciable deformation, particularly if thin-walled instruments were suspended in the furnace.

A. Cousen.

Heat treatment as an agent in [quartz] rock breaking. B. W. Holman (Bull. Inst. Min. Met., Jan., 1927, advance copy, 16 pp.).—When many forms of quartz are heated at 560—600° and quenched in water the mineral becomes so friable that it may be reduced to a 30-mesh powder by gentle pressure between finger and thumb. The best results are obtained by rapid heating, but equally good results may be obtained by repeated heating to 300—400° followed by quenching in water. Air-cooling between the successive heatings results, however, in a noticeable increase of compressive strength. The fact that a marked change takes place in the dielectric constant, and that the heated mineral is more readily

soluble in hydrofluoric acid indicates that the quartz undergoes molecular re-arrangement; this change begins at about 340°, and is complete above 575°. The fuel consumption of a large-scale plant using a rotary coal-fired kiln would be about 40 lb. of coal per ton. It is suggested that the above described phenomenon formed the basis of the Roman method of "fire-setting."

A. R. Powell.

Hydraulic properties of a slag sand. A. Guttmann and C. Weise (Zement, 1926, **15**, 527—531; Chem. Zentr., 1926, II, 1783).—Dumped slag-sand loses its granular hard structure and cakes together somewhat. It smears on rubbing and produces, in iron–Portland cement, greater strength than does fresh material. The vitreous sand, on storing, forms silicic acid, and the porous particles there existing adsorb litmus and methylene blue. The formation of a gel is indicated by the dehydration curve. In storing it is partly converted into the carbonate. Water which contains carbon dioxide decomposes fresh slag in 3 days, so that it can be coloured with methylene blue. The ability of the slag to react can be determined by attacking finely-powdered sand with 5% potassium hydroxide solution, then washing, and colouring with methylene blue. The slag is also mixed with standard sand and, after a 3-day treatment with water, tested for resistance to compression; the material which gives the strongest colour gives the highest resistance to compression. A 28-day treatment gives similar results. The method does not give such definite results if a mixed storage is substituted for the water storage.

A. Cousen.

Determination of alumina in silicate analysis. F. Steinbrecher (Sprechsaal, 1926, **59**, 295—297; Chem. Zentr., 1926, II, 1783).—In the method of Kallauner and Matejka some silica is always precipitated with the iron and aluminium; this may be removed by evaporating four times with sulphuric acid and 2 drops of hydrofluoric acid. The process occupies 30 min., and no iron or aluminium is lost by volatilisation.

A. Cousen.

Study of the firing and glazing of porcelain and stoneware by measuring the porosity. P. Bremond (Céramique, 1926, **29**, 217—226; Chem. Zentr., 1926, II, 1783).—Glazing usually takes the same course, felspar and lime generally being present. Felspar porcelain commences to glaze at 1050°, lime porcelain at 1200°, the degree of glazing being equal at 1250°. Stoneware glazes sooner than porcelain, and its porosity can be kept lower (with correct firing-control) when the same firing conditions hold, if the enclosed gas particles are given time to escape. From 1200° carbon may be left in the ware through dissociation of carbon monoxide, but this can be oxidised again by carbon dioxide and water. To effect these reactions the porosity must be less than 5%. By regulating the furnace atmosphere and the temperature either the oxidising or the reducing condition may be obtained, and any desired degree of porosity produced. The presence of sulphides and fluorides can seriously increase the porosity of the material.

A. Cousen.

Trend of design in modern coke-oven construction and its bearing upon refractory materials. Vickers and Green.—See II.

Patents.

Manufacture of glass or quartz transparent to ultra-violet light. R. Haddan. From Corning Glass Works (E.P. 263,410, 1.7.26).—A glass transparent to light waves shorter than 300 $\mu\mu$ can be prepared by reducing the iron impurities in the raw materials to less than 0·055% as ferric oxide, and the titanium oxide impurities to less than 0·050%, and carrying out the melting in a crucible free from these oxides, under non-oxidising conditions, *e.g.*, in the presence of carbon, carborundum, or hydrogen. Calcium phosphate glass, silicate glasses, and fused quartz can be prepared in this way.

B. W. Clarke.

Apparatus and process for fusing silica. British Thomson-Houston Co., Ltd., Assees. of P. K. Devers (E.P. 255,118, 13.7.26. Conv., 13.7.25. Cf. E.P. 240,489; B., 1926, 667).—Quartz is fused under a vacuum in a closed crucible of porous, highly refractory material, such as graphite, provided with a heavy-walled exhaust pipe of similar material, through which the gaseous products of the reaction are withdrawn, thereby preventing contamination of the fused quartz.

B. W. Clarke.

Kilns for salt glazing. W. A. Robertson (E.P. 261,262, 21.4.26).—The kiln comprises a cylindrical wall provided with fire holes accommodating furnaces placed opposite to bag walls fitted to a perforated bottom, the products of combustion passing downwards through this, and then, by means of up-take vents regulated by dampers, over the crown of the kiln, which is surmounted by a coned chimney.

B. W. Clarke.

Forming sheet glass. L. Mellersh-Jackson. From Hartford-Empire Co. (E.P. 263,394, 31.5.26).

IX.—BUILDING MATERIALS.

Patents.

Fine grinding of cement or the like in ball or tube mills. F. L. Smidth & Co., A/S., Assees. of F. L. Smidth & Co. (E.P. 256,987, 13.8.26. Conv., 14.8.25).—Ball or tube mills are fitted with a tube which rotates with the mill, and has a worm conveyor and an outlet towards the discharge end of the mill fitted with scoops which conduct the balls into the tube; in this they are mechanically or chemically cleaned from the adhering film of finely-ground cement, which reduces their grinding action, and returned to the mill at the supply end.

B. W. Clarke.

Manufacture of spontaneously pulverised aluminous cements. U. B. Voisin (E.P. 259,203, 23.9.26. Conv., 2.10.25).—Raw materials for the manufacture of aluminous cement are so selected that the calcined mass contains at least 25% of alumina, together with 40—50% of that amount of silica, and a lime content of 3—3·25 times the silica content. Spontaneous pulverisation of the mass will then occur upon slow cooling, thereby eliminating the high cost of grinding.

B. W. Clarke.

Process and apparatus for the preparation of raw materials used in the manufacture of cement. I. E. Lanhoffer (E.P. 263,221, 19.9.25).—The raw materials are formed into a paste of suitable plasticity, which is squirted in narrow strips on to a conveyor

in a current of hot gases, where the strips are partially cut through, so that when the material is delivered to a rotary drum the strips break themselves up into fragments of practically spherical form. These fragments are dried at a low temperature to prevent deformation, freed from dust, reheated, and fed into a rotary cement kiln. The use of small uniform fragments of this type results in greatly increased thermal efficiency in the kiln.

B. W. CLARKE.

Manufacture of porous or cellular cements. J. A. RICE (E.P. 263,571, 19.10.25).—A foam-assisting agent, *e.g.*, natural or artificial resins, glycerol, cellulose or rubber derivatives, in solution or suspension in suitable liquids such as acetone or alcohol, with or without the addition of formalin, is added to a foam-producing agent consisting of a colloidal solution of glue, gelatin, or glucoside- and saponin-containing materials, with or without the addition of soap or formalin, and the mixture is beaten to form a foam and added to cement, mortar, etc. to form a strong porous or cellular concrete on setting.

B. W. CLARKE.

[Lime-]mixing apparatus. F. B. LEOPOLD (U.S.P. 1,613,663, 11.1.27. Appl., 6.6.25).—A lime-treating apparatus comprises a hydrating chamber and a solution chamber, each provided with a mixing device and a lime-feeding device. A water motor drives these devices in one of the chambers, the escaping water being conducted to the chambers.

B. W. CLARKE.

Apparatus for making plaster and mortar. A. E. TUTTLE, Assr. to BLUE DIAMOND MATERIALS Co. (U.S.P. 1,613,682, 11.1.27. Appl., 14.11.24).—Lime putty is pumped from a suitable container through a delivery pipe, whence it issues in a continuous jet, the length of which is measured, and determined by a regulating valve in the delivery pipe.

B. W. CLARKE.

Composition for making casting patterns, pattern mounts, and the like. C. H. PAPÉ (U.S.P. 1,614,315, 11.1.27. Appl., 26.9.23).—A slow-setting cementitious composition comprises a mixture of comminuted magnesium chloride with fibrous material and a dilute solution of magnesium chloride.

B. W. CLARKE.

Improving the quality of wood. A. J. AUSPITZER (E.P. 253,925, 17.6.26. Conv., 17.6.25).—Soft wood held in a suitable container is subjected to a drawing or rolling process in a longitudinal direction, thereby producing a consolidation and hardening of the wood without destroying the cohesion of the fibres.

B. W. CLARKE.

Preserving wood by impregnation. J. HIMMELSBACH, Assr. to GEBR. HIMMELSBACH A.-G. (U.S.P. 1,613,459, 4.1.27. Appl., 31.3.26. Conv., 3.11.25).—See E.P. 253,041; B., 1926, 669.

Bitumen emulsion (E.P. 263,307).—See III.

Composition of bitumen and rubber (E.P. 263,028).—See XIV.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Blast-furnace process. P. W. UHLMANN (Chem.-Ztg., 1927, **51**, 37—38).—The heat balance of a suggested new blast-furnace process for the production of pig-iron using gaseous or liquid fuel has been calculated, and it is shown that, on theoretical grounds, it should be possible economically to produce a very pure iron direct from ore using a mixture of equal volumes of producer gas and electrolytic hydrogen together with air enriched by oxygen for firing the blast furnace and for reducing the ore. The process should be cheaper than direct electric smelting in spite of the necessity of obtaining the hydrogen and oxygen required by the electrolysis of water. In place of producer gas, crude petroleum, atomised tar spray, or other liquid fuel may be used together with charcoal or anthracite to carburise the iron sufficiently to make it flow freely from the furnace. This process has the advantages that contamination of the iron with the sulphur and phosphorus impurities usually present in the solid fuel used in blast-furnace smelting is avoided, and the composition of the iron and temperature of the furnace can be more easily controlled.

A. R. POWELL.

Cooling devices for the Siemens–Martin furnace. G. BULLE (Stahl u. Eisen, 1927, **47**, 41—52, 85—90).—A large number of cooling methods applicable to various parts of the furnace are described. The advantages obtained through cooling are enhanced durability of furnace with consequent increase in service and less frequent interruptions and repairs to flame passage. The disadvantages are the increased fuel consumption and cost of installing and operating the cooling plant.

L. M. CLARK.

Uses of coke-oven gas in the steel industry. L. M. WILSON (Fuel, 1927, **6**, 29—36).—Coke-oven gas can be used in melting furnaces designed for producer gas by introducing some of the extra air required for combustion as primary air. A short sharp flame very suitable for melting purposes is produced. Coke-oven gas is not recommended for use in soaking pits, but may be used with advantage in slab reheating furnaces if the latter are specially designed for the purpose. It is ideal for continuous furnaces. By operating with a mixture of coke-oven gas and producer gas and varying the relative proportion to meet the varied requirements at different stages of steel making, considerable heat economy may be achieved. Factors governing the type of flame produced are discussed. "Flame intensity" depends on the calorific value of the gas–air mixture and the velocity constant of the reactions in progress; in furnace work the latter is mainly influenced by the amount of turbulence. Surface combustion due to contact catalysis also plays a part, particularly with gases of high hydrogen content, and may produce harmful effects on the brickwork. Very great economy can be attained where coke ovens, blast furnaces, and steel works are in close proximity, by using a mixture of coke-oven gas and blast-furnace gas (giving preferably about 250 B.Th.U. per cub. ft.) instead of producer gas. The necessary modifications in plant and operation which would be occasioned by the change are discussed.

A. B. MANNING.

Deep cementation by gas and its influence on the core in case-hardened steel. W. ROHLAND (Stahl u. Eisen, 1927, **47**, 52—57).—By the action of coal gas at

900—1050°, on two carbon steels (C 0·06%, 0·12%) and on a steel containing 1·85% Ni, 0·44% Cr, and 0·18% C, a case-hardening effect was produced to a depth of 1·55—5·55 mm. A case 4 mm. thick was obtained from which free cementite was absent, and which was made glass-hard throughout. A double quenching from 930° in oil and from 770° in water produced a fine-grained, ductile core in the carbon steels, though the grain size was thereby raised to between two and three times its original value. The nickel steel gave more satisfactory results, its grain size being increased only by 50%, whilst its higher tensile strength was combined with a sufficient degree of ductility.

L. M. Clark.

Changes of volume on cold-working steel. E. Houdrement and E. Bürklin (Stahl u. Eisen, 1927, **47**, 90—93).—Contrary to Maurer's theory of hardening steel, production of the same change in density by cold working and by quenching gives greater hardness and higher tensile strength in the latter case. It is concluded that both types of hardening are due to the development of internal stresses, which, in turn, give rise to change in volume. The fact that the density changes accompanying equal increases in strength are not the same in the two cases is to be attributed to the difference in stress distribution, which is extremely uniform in quenched steel but most irregular in cold-worked specimens. L. M. Clark.

Impact tests of steels at low temperatures. R. Yamada (Sci. Rep. Tôhoku Imp. Univ., 1926, **15**, 631—659).—Bending impact (Charpy machine), tension impact, and repeated impact tests have been made on simple and alloy steels at temperatures down to that of liquid air. Very low carbon steels (0·1% C) bend and do not break under the Charpy test at room temperature, but break below a certain temperature, which depends on the size of the notch in the test piece. Steels with more than 0·3% C are broken even at room temperature. All steels become more brittle with fall in temperature, but in both plain and alloy steels a sorbitic steel is not merely more tough than a pearlitic steel at room temperature, but shows a less rapid decrease in toughness as the temperature falls. Pearlitic steels containing nickel and chromium are similarly superior to plain carbon steels, but small quantities of copper or manganese have little effect. If the absorbed energy is plotted against the bending angle a straight line is obtained passing almost through the origin, indicating that the absorbed energy is used almost entirely in bending the test-piece. The sorbitic structure is again superior in the repeated impact tests. Microscopical examination showed that in ferrite grains the fracture takes place through the crystal at room temperature, but intercrystalline fracture occurs to an increasing extent as the temperature falls. Pearlite grains are broken in all directions relative to that of the lamellæ.

W. Hume-Rothery.

Duralumin. R. Beck (Z. Metallk., 1927, **19**, 12—14).—The characteristic properties of duralumin alloys are briefly reviewed, and a table showing average values of the chief mechanical properties of the three alloys that are used in Germany for constructional work is given, together with examples of their use in boat-building and in cotton-spinning machinery.

A. R. Powell.

Spectrographic detection and determination of impurities in aluminium and its alloys. R. Adan (Bull. Soc. chim. Belg., 1926, **35**, 447—451).—Photographs of arc and spark spectra are examined, and the presence and proportions of the common impurities deduced from the presence and strength of the chief lines. The most persistent lines for copper, lead, zinc, tin, iron, magnesium, and silicon are given.

S. I. Levy.

Aluminium as constructional material in the organic chemical industry. Buschlinger (Z. Metallk., 1927, **19**, 25—36).—The behaviour of a large number of organic chemical products in contact with metallic aluminium (99·4%) at different temperatures is recorded. The paraffin and aromatic hydrocarbons, lubricating oils, crude petroleum, trichloroethylene, and carbon tetrachloride have little or no action either hot or cold, but ethylene dibromide vigorously attacks the metal at high temperatures. Aluminium is slightly attacked by ethyl alcohol–water mixtures containing more than 20% of alcohol, and more vigorously by methyl alcohol; aluminium vessels, however, are suitable for use in brewing operations conducted at temperatures below 50° and for storage purposes. The action of the lower fatty acids, oxalic, succinic, citric, and tartaric acids is very slight at the ordinary temperature, but increases rapidly with the temperature; the action of tartaric acid is more vigorous the lower the concentration. Phenols are without action up to about 100°, but at 140° anhydrous phenols rapidly attack the metal. A. R. Powell.

Aluminium in electro-technology. W. Wunder (Z. Metallk., 1927, **19**, 36—37).—For the same carrying capacity as copper an aluminium conductor must have a cross-section 1·6 times as large and a diam. 30% greater, but the weight of the conductor is only half that of the corresponding copper wire. A core of steel having one-sixth the cross-section of the aluminium is necessary when large conductors are carried over long spans. Difficulties also arise at the supports owing to corrosion set up by the *P.D.* between the wire and the support. Aluminium alloys may, however, be used for the housing of electrical units, especially those which are portable. A. R. Powell.

Determination of tin and lead in solder and of tungsten in ferrotungsten by calculations on the Archimedean principle. Koch (Chem.-Ztg., 1927, **51**, 54—55).—By determining the sp. gr. of solder or of ferrotungsten by the water-displacement method, the proportions of tin and lead or of tungsten present may be calculated from the known sp. gr. of tin, lead, tungsten, and iron. As the sp. gr. of manganese and tin are so near to that of iron, the proportion of these impurities in ferrotungsten, if small, has no effect on the tungsten calculation. A. R. Powell.

Lixiviation of copper minerals with ammonia and preparation of arsenical compounds of copper. G. Fester and F. Bertucci (Anal. Asoc. Quím. Argentina, 1926, **14**, 243—250, and Z. angew. Chem.,

1927, **40,** 82—84).—By repeated extraction of finely-divided cuprite with 10% ammonia, up to 94·3% of the copper present was extracted. The recovery of copper is less where some is present as sulphide or silicate; a smaller recovery is obtained from roasted ores. Cupric oxide may be obtained by passing steam through the ammoniacal solution. Electrolytic separation was unsatisfactory. The copper may also be precipitated as copper arsenate by addition of arsenic acid, or as copper arsenite and arsenate by adding arsenious oxide and passing steam and air through the solution. G. W. Robinson.

Electromagnetic separator for laboratory use. C. J. Ksanda (J. Opt. Soc. Amer., 1926, **13,** 713—715).—A powerful electromagnet is described by means of which minerals may be fractionated with a high degree of separation, according to their magnetic susceptibilities. Each pole is wound with 5500 turns carrying 1·95 amp. and operated on 110 volts. Full constructional details are given. C. J. Smithells.

Patents.

Manufacture of pure iron. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 262,938, 14.12.25).—Iron carbonyl vapour, formed by the action of carbon monoxide at a high temperature and pressure on impure iron or iron ores, is diluted either by admixture with an inert or reducing gas and/or under reduced pressure, and then passed over or through substances heated at or above 100°, *e.g.*, iron turnings or molten metals, oils of high b.p., or melts of anthracene, phenanthrene, or paraffin hydrocarbons of high mol. wt. The rate at which the iron carbonyl vapour passes over should be such that no unaltered vapour escapes with the waste gases. The iron so obtained is substantially free from carbon. The carbon monoxide may be recovered completely. M. E. Nottage.

Magnetic materials [iron-nickel alloys]. Western Electric Co. From Bell Telephone Laboratories, Inc. (E.P. 263,207, 18.8.25. Cf. E.P. 245,986; B., 1926, 244).—The permeability in magnetic fields of small intensity and the resistivity of iron-nickel alloys containing from 70 to 85% Ni may be substantially increased by the addition of a small proportion of a third element such as chromium, when the alloy is subsequently suitably heat treated. For an alloy containing 78·5% Ni, 2% Cr, and remainder iron, the best magnetic properties are obtained by heating at 1100°, slowly cooling, and reheating to the neighbourhood of the magnetic transition temperature, followed by rapid cooling below this transition point. L. M. Clark.

Production of an aluminium solder. O. R. Croy (E.P. 263,402, 15.6.26).—An aluminium solder producing strong joints with any solderable metal is obtained by the addition of 0·1—5% Al to a molten mixture of 70—25% Zn and 25—70% Sn. The solution of the aluminium is carried out under a layer of charcoal, ammonium chloride being used as a flux. L. M. Clark.

Production by electro-thermic means of aluminium-silicon alloys practically free from carbide. Metallbank u. Metallurgische Ges. A.-G. (E.P. 252,160, 10.5.26. Conv., 12.5.25).—By using initial materials free from iron, an aluminium-silicon alloy which is almost free from iron may be produced, whereas if the silicon content is increased to 35—47%, then, by suitably proportioning the ingredients of the furnace charge, the formation of carbide disappears almost completely. These alloys rich in silicon may be alloyed with pure aluminium with or without the incorporation of other known additional metals. M. E. Nottage.

[Lead] plating method. R. J. Shoemaker, Assr. to Leadizing Co. (U.S.P. 1,608,250, 23.11.26. Appl., 13.7.23. Cf. U.S.P. 1,405,167 and 1,566,984; B., 1922, 221 A; 1926, 163).—Iron and steel articles can be given a dense, smooth, adherent coating of lead by first dipping them in molten lead, or in a 45% solution of lead acetate at about 75°, and then adding further lead by electrolysis of a 10% lead acetate solution. In depositing copper, tin, nickel, or silver on iron by electrolysis, it is of advantage first to dip the article in molten lead or lead acetate. T. S. Wheeler.

Sintering apparatus. J. E. Greenawalt (E.P. 263,363, 6.4.26. Conv., 5.2.26).—A mechanism is described for emptying the sintering pan used in ore sintering operations, by complete rotation through 360°, and at the same time for producing a rocking motion in the attached grate bars. L. M. Clark.

Metal coatings on aluminium and aluminium alloys. Dr. O. Sprenger, Patentverwertung Jirotka m.b.H., Assecs. of B. Jirotka (U.S.P. 1,607,676, 23.11.26. Appl., 15.1.26. Conv., 29.10.24).—See E.P. 249,971; B., 1926, 496.

Apparatus for determining the moisture content of moulding sand (E.P. 263,450).—See I.

Coating vessels etc. to render them chemically stable (E.P. 250,525).—See XIII.

XI.—ELECTROTECHNICS.

Control of oils in transformers. Tilgner.—See II.

Valuation of pitch as a binding material for carbon electrodes. Schuchardt.—See III.

Submarine insulation with special reference to the use of rubber. Williams and Kemp.—See XIV.

Patents.

Apparatus [anodes] for electro-deposition. Internat. Copperclad Co., Assees. of T. Robinson (E.P. 255,113, 4.8.25. Conv., 5.8.24).—An anode assembly for use in electro-deposition, more especially in cases where the plating or the article itself is relatively thin, comprises an anode on which is mounted an insulating spacing member having portions lying against each face of the anode, and a contact member also carried by the anode and having portions lying on the outer faces of the spacing member. J. S. G. Thomas.

[Electrode] for the electrification of gases [for producing ozone]. C. H. Shearman (E.P. 263,356, 13.3.26).—Groups of points are arranged in a plane or planes parallel to the electrode and around regularly-spaced apertures in the electrode, so that the distance

between the nearest points of a group is $\frac{1}{4}$—$\frac{1}{8}$ of the distance, measured in the direction of gas flow, between the nearest points of two groups in one plane.

J. S. G. Thomas.

Transmitting electric charges through a gaseous medium. L. Levy (E.P. 252,167, 11.5.26. Conv., 14.5.25).—Electric charges are propagated in a gaseous fluid by means of carrier waves or beams, *e.g.*, acoustic or ultra-acoustic waves of frequencies from 1000—10,000 periods/sec. J. S. G. Thomas.

Thermionic valve [cathode]. A. C. Hyde (U.S.P. 1,608,317, 23.11.26. Appl., 26.5.25. Conv., 20.6.24).—A colloidal solution of tungstic acid, containing finely-divided thorium or uranium, is coated on a core of tungsten or molybdenum, which is then dried and heated *in vacuo*, or in an inert atmosphere, at 1700—2000°, when a highly emissive coating is obtained on the core. In place of tungstic acid, any salt of that metal, or of a metal such as platinum, which will yield the metal on heating and which, preferably, will dissolve in an organic solvent, can be employed. T. S. Wheeler.

Tungsten arc lamp. General Electric Co., Assees. of Patent-Treuhand-Ges. für Elektrische Gluhlampen, m.b.H. (E.P. 263,405, 19.6.26. Conv., 3.2.26).—In a direct current tungsten arc lamp, two or more anodes, connected in parallel, co-operate with a single cathode, and are connected in series with a resistance. A slit is formed between adjacent anodes oblique to the common plane, so that the anodes in use present an unbroken illuminated surface in the principal direction of radiation. A small quantity of a gas of low anode drop of potential, *e.g.*, 2—10% of argon or mercury, is added to the nitrogen used as filling.

J. S. G. Thomas.

[Electrolyte for] electric batteries. F. Melms (E.P. 263,587, 6.11.25).—Alkali-cellulose is used as an electrolyte in batteries and accumulators.

J. S. G. Thomas.

[Retaining material in] electric storage batteries. W. B. Stone (E.P. 263,024, 19.5.26).

Breaking petroleum emulsions (U.S.P. 1,612,180). —See II.

Aluminium in electro-technology. Wunder.—See X.

XII.—FATS; OILS; WAXES.

New index for the determination of butter fat. F. von Morgenstern (Z. Unters. Lebensm., 1926, **52**, 385—388).—The fat is saponified with potassium hydroxide and glycerin, and the solution diluted with water (cf. B., 1926, 447). After cooling, saponified cacao butter and copper sulphate solution are added, the whole is shaken, and after settling is filtered from the insoluble fatty acid copper salts. The filtrate is acidified with dilute sulphuric acid, distilled, and the distillate titrated with 0·1N-sodium hydroxide. From an empirical table the percentage of butter fat may be read off from the titration figure, which is called the "copper index."

F. R. Ennos.

Susceptibility of fats to autoxidation. G. E. Holm, G. R. Greenbank, and E. F. Deysher (Ind. Eng. Chem., 1927, **19**, 156—158).—Loosely bound oxygen compounds are regarded, especially in butter oils, as the first compounds formed in the autoxidation process, and under certain conditions, such as long storage and action of light, their potential oxidising capacity may affect oxidation. Conditions of manufacture in the refining process, rather than their constitution, appear to affect the susceptibility of cotton-seed oils to oxidation, whereas acids have relatively little effect. The oxygen-absorption reaction is probably not entirely analogous in this case to the reaction involved in autoxidation of the oleic acid radical. Hydroxyl groups attached to molecules other than that containing an unsaturated linking have a retarding effect on autoxidation, apparently due to a reaction with some intermediate oxidising compounds, but once absorption begins it seems to proceed at the rate of the original oil or fat. D. G. Hewer.

Hydrogen electrode in the study of the rate of saponification of oils and fats by aqueous alkali. J. W. McBain, H. S. Howes, and (Miss) M. Thorburn (J. Physical Chem., 1927, **31**, 131—143. Cf. Norris and McBain, B., 1922, 719 A; Langton, *ibid.*, 825 A).—The rates of saponification of coconut oil, trilaurin, and tripalmitin have been measured by a rapid and convenient method involving the use of a hydrogen electrode, and by means of which the influence of all factors can be separately studied. The rate is proportional to the concentration of hydroxyl ions as measured by the hydrogen electrode. The first small amount of oil greatly increases the rate, but further additions have a comparatively small effect, agreeing with Norris and McBain (*loc. cit.*) that a given amount of soap has a limited emulsifying action. Increasing the initial amount of soap present rapidly increases the rate constant up to a concentration of $2N_w$-soap, beyond which the reaction mixture becomes too viscous for this method to be used. The explanation given is that the oil is more perfectly emulsified when more initial soap is present. The effect of salt depends on the physical state of the system, but the fastest rates were obtained when the concentration of sodium chloride approached saturation. Addition of glycerol diminishes the rate. Tripalmitin is more rapidly saponified than coconut oil. L. S. Theobald.

Gas coagulation theory of the drying of fatty oils. L. Auer (Kolloid-Z., 1926, **40**, 334—338).—The experiments were conducted by measuring the increase in weight of varnish in air and in carbon dioxide contained in desiccators of varying drying power and at different pressures. The curve representing the increase in weight of the oil on exposure to air as a function of time can be resolved into two independent curves representing the increase due to absorption of oxygen and that due to the absorption of water vapour. In a high vacuum (10^{-6}mm.) the oil remained unchanged after many months. Drying occurs in gases other than air or oxygen, and is supposed to be due to a true coagulation after absorption. E. S. Hedges.

Highly unsaturated fatty acids of fish oils. II. Limit of unsaturation in menhaden oil. R. R. McGregor and G. D. Beal (J. Amer. Chem. Soc., 1926, **48**, 3150—3161. Cf. A., 1923, i, 647).—Methyl esters were prepared from 12 litres of menhaden oil, fractionated,

the constants of the fractions determined, and the amounts of different carbon chains present in certain fractions determined by Twitchell's method of hydrogenation and m.p. depression (cf. A., 1917, ii, 428). Certain fractions of the esters were brominated in ether at $-10°$, and the ether-insoluble bromides extracted with boiling bromobenzene, which effected a partial separation, as the more highly brominated esters remained undissolved. From the results obtained, compositions of certain ester fractions and their bromides were deduced as follows:—Fraction 5 [b.p. 160—170°/3 mm., iodine value (Wijs) 146·3, mol. wt. 277·2, $n^{19.5}$ 1·4630]: C_{16} acids 12·1%, C_{18} acids 72·5%, C_{20} acids 15·4%, bromides insoluble in bromobenzene (Br = 66·4%) chiefly hexa- and octa-bromostearates; fraction 7 (b.p. 180—190°/3 mm., iodine value 325·0, mol. wt., 305·6, $n^{19.5}$ 1·4894): C_{18} acids 1·1%, C_{20} acids, 70·3%, C_{22} acids, 28·6%, insoluble bromides (Br = 71·46%), decabromoarachidate; fraction 8 (b.p. 190—200°/3 mm., iodine value 348·8, mol. wt. 319·5, $n^{19.5}$ 1·4962): C_{20} acids, 8·6%, C_{22} acids, 91·4%, insoluble bromides (Br = 71·99%) chiefly methyl dodecabromobehenate. It is concluded that the highest unsaturation to be found in menhaden oil corresponds to 2 double linkings in the C_{14}, 3 in the C_{16}, 4 in the C_{18}, 5 in the C_{20}, and 6 in the C_{22} acids, which is in accordance with the results of Armstrong and Hilditch (B., 1925, 137, 458). F. G. WILLSON.

Flower waxes. J. F. S. STRAMAN (Pharm. Weekblad, 1927, 64, 52—69).—The waxes examined were obtained during the manufacture of perfumes from the fresh flowers. The treatment consisted of extraction of the flowers with light petroleum; after removal of the solvent, the residues were treated with alcohol for removal of the odorous ethereal oils, the waxes remaining undissolved. Samples from roses, jasmine, mimosa, Parma violets, and hyacinths were examined. Microscopical examination of the petals showed the waxes to be present as outer protective coverings of the epidermis. The results of chemical examination were as follows:—

Wax	Violet	Mimosa	Rose	Hyacinth	Jasmine
d^4	0·963	0·960	0·929	0·956	0·932
m.p...	58°	59°	57·5°	48°	60°
Acid value	12·01	16·42	3·40	2·20	5·40
Ester value ..	53·68	89·31	23·98	102·66	55·50
Saponif. value ..	65·69	105·73	27·38	104·86	60·90
Iodine value ..	17·28	34·58	12·87	55·76	40·26
Composition—					
Hydrocarbons ..	46·95	51·90	74·20	25·42	49·85%
Higher alcohols	10·25	23·90	11·70	25·02	14·35%
Saturated fatty acids	34·99	14·20	?	27·66	21·31%
Unsaturated fatty acids	5·80	8·00	?	19·14	14·50%

The hydrocarbons in each case seemed to consist almost entirely of hentriacontane, $C_{31}H_{64}$, the m.p. and mol. wt. being reasonably close to those of this material; the analyses in all cases gave low results. The higher fatty acids consist chiefly of cerotic acid, and of the lower acids only acetic acid could be identified. Mimosa wax and hyacinth wax also probably contain palmitic and stearic acids, but no volatile acids. The fatty acids from the rose wax were insufficient for separation, but only traces of unsaturated acids appeared to be present. S. I. LEVY.

Xylene and xylene percentage numbers in butter. VAN RAALTE.—See XIX.

PATENTS.

Fat-splitting agent (U.S.P. 1,608,341, 23.11.26. Appl., 12.12.24. Conv., 5.1.24).—See E.P. 227,089; B., 1925, 728.

Dispersing substances in water (U.S.P. 1,609,308).—See I.

Cleanser for grease etc. (E.P. 263,275).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Boiling of linseed oil. J. S. LONG, C. A. KNAUSS, and J. G. SMULL (Ind. Eng. Chem., 1927, 19, 62—65).—The observed rates of change of hexabromide value, iodine value, and mol. wt. of linseed oil when heated at 293° *in vacuo* or in a stream of nitrogen show that in the first stages intramolecular rearrangements preponderate, leading to decrease in the number of $\Delta°$ ethenoid linkages with little corresponding change in mol. wt. Mono- and di-glycerides thus or otherwise formed may then undergo condensations involving elimination of water etc., causing the subsequent relatively large increases in mol. wt. If the oil be heated in the presence of sulphur, selenium, or tellurium, condensation reactions are almost entirely absent. Linolenic monoglyceride was synthesised by heating linolenic hexabromide, zinc dust, and glycerol, and showed the anticipated exceptionally rapid rise in mol. wt. on heating. S. S. WOOLF.

Chemistry of tung oil. F. H. RHODES and C. J. WELZ (Ind. Eng. Chem., 1927, 19, 68—73).—On bodying tung oil at 150°, 170°, 190°, and 220°, the iodine value falls, the acid value and the saponif. value are not appreciably affected, whilst the mol. wt. rises until gelatinisation occurs at a value practically independent of the temperature of heating. The sample under test gelatinised at a mol. wt. of approx. 1700. Tung oil, gelatinised at 220°, was fractionally extracted with boiling light petroleum and absolute alcohol, and the fractions were separately examined. The soluble portion of the gel is shown to be a dipolymeride, containing one tetramethylene ring, formed by combination of 2 mols. of elæostearin at a single pair of double linkages. The remainder of the gel is more highly polymerised, in a manner not at present fully explicable. This holds for oils gelatinised at relatively low temperatures only, gels produced at 360° being contaminated by decomposition products of low mol. wt. The heat of gelatinisation of tung oil, determined by a new method involving its utilisation to vaporise weighed amounts of naphthalene, corresponds to a coagulation of polymerised material already present in the thickened oil, rather than to the formation of a fresh insoluble compound. The oxygen-absorbing power of tung oil decreases on bodying at a rate apparently dependent on the formation of the highly polymerised substance. It is, therefore, considered that the dipolymeride formed in the early stages of bodying is subsequently enmeshed in the gel produced when the progressively developed complex polymeride reaches its coagulation concentration. S. S. WOOLF.

Relative method for determining particle size of pigments. G. F. A. STUTZ and A. H. PFUND (Ind. Eng. Chem., 1927, 19, 51—53).—An apparatus for

measuring the intensity of light transmitted by suspensions of a pigment at different states of subdivision is described. It is shown that the opacity of a pigment attains a maximum at a definite particle size, characteristic of the particular type of material. Suitable calibration curves having been prepared, the average particle size of any suspension may be rapidly determined. S. S. Woolf.

Relation of yield value to particle size. H. Green and G. S. Haslam (Ind. Eng. Chem., 1927, **19**, 53—57).—The flocculated structure which gives rise to the "yield value" of a plastic mixture is attributed to interfacial tension and quasi-frictional resistance between layers. Expressions for interfacial area and number of points of contact per unit area of one layer of particles are derived, and the relations between these values, the percentage weight and volume of pigment, and yield value of experimental mixtures are represented graphically and discussed. Yield value is also correlated with the various average diameters of the particles of a non-uniform material. S. S. Woolf.

Yellow ink for marking rubber tensile strips. C. G. Cashion (Ind. Eng. Chem., 1927, **19**, 45).—An ink prepared by precipitating lead chromate by potassium chromate, boiling, filtering, and grinding the lead chromate into glycerol before it is quite dry, and using on a blank pad moistened to the right consistency with glycerol, gives satisfactory lines for stress work, and is specially suitable for black treads and in ageing work. D. G. Hewer.

"Acrolite"—a new synthetic resin. J. McIntosh (Ind. Eng. Chem., 1927, **19**, 111).—"Acrolite" is the condensation product of glycerol and excess of phenol. It is said to be superior to other commercial artificial resins in lustre and heat-resisting capacity, and to have all the desirable properties of the phenol–formaldehyde type of synthetic resin. S. S. Woolf.

Determination of colour intensity of resins, varnishes, oils, etc. E. Fonrobert (Farben-Ztg., 1927, **32**, 795—797, 850—851).—The author enumerates the advantages of exact determinations of colour of resins, varnishes, oils, etc. in connexion with the evaluation of raw materials, control of factory processes, and maintenance of uniform finished products. The Fonrobert–Pallauf colorimeter detailed is a simpler modification of the previously described apparatus for carrying out the determination of "iodine colour numbers" (cf. B., 1926, 201). Relatively large variations are shown to exist in the colour of commercial samples of the various rosin grades. S. S. Woolf.

Effective metal content in siccatives and its determination. P. Müller (Farben-Ztg., 1927, **32**, 798—800, 851—853).—A true measure of the efficiency of a siccative preparation is not afforded by the customary ash determination, since this records uncombined metals, *i.e.*, unfixed oxides (fused driers) or excess of precipitating agents (precipitated driers) as well as the active metal content, *i.e.*, that combined with resin- or fatty oil-acids. From the observed drying times of a series of linseed oil–driers mixtures heated for ½ hr. at 110—120°, the siccative action of suspended or unfixed driers is shown to be very small, and any apparent effect is to be ascribed to local overheating, with consequent fixation, rather than to catalytic action. A suggested method for resolving the metal content of driers into effective and inactive portions is as follows :—The resinates, linoleates, etc. are extracted by shaking in the cold with 3—4 vols. of acetic anhydride, in which solvent the majority of unfixed oxides and salts likely to be present are insoluble. The solution is evaporated to dryness, and the resinous residue converted into metal sulphates by means of concentrated sulphuric acid and ashing. This sulphated residue can be quantitatively analysed with more reliability than the normal ash—a mixture of reduced metal and oxides in doubtful state of oxidation. The drier as a whole and, if necessary, the portion insoluble in acetic anhydride can be similarly sulphated, and the active proportion calculated. S. S. Woolf.

Water content of bleached shellac, and a water-containing artificial shellac. F. Baum (Chem.-Ztg., 1927, **51**, 42—43).—The price of bleached shellac is often lower than that of natural shellac, notwithstanding the loss of material by alkaline hypochlorite bleaching. Such shellac contains 25—30% of water, probably as a fine mechanical separation. A similar phenomenon occurs in an artificial shellac, which is a dark reddish-brown, transparent resin not affected by bleaching. A considerable proportion of the coloured portion can be separated by treatment with alcohol, benzene, and water. W. G. Carey.

Particle size. Lukirsky and Kosman, also Calbeck and Harner.—See I.

Thermochemical comparison of various types of lamp black. Hock and Bostroem.—See II.

Gas coagulation theory of the drying of fatty oils. Auer.—See XII.

Patents.

Coating vessels etc. to render them chemically stable. Säureschutz Ges.m.b.H. (E.P. 250,525, 10.9.25. Conv., 11.4.25).—Metal sheets, tubes, or other apparatus to be rendered proof against acid etc. are provided with holes, notches, or recesses, thus giving good adhesion of a layer of phenol–aldehyde condensation product, which is applied and subsequently heat-"cured." With or without a preliminary removal of the outer crust of the first hardened layer, and a further heat treatment to drive off any volatile constituents still remaining, further layers of phenol–aldehyde resin, admixed with chemically-stable fillers, and fabric inserts may be applied. S. S. Woolf.

Cleansing articles contaminated by oil, grease, paint, etc. J. L. Biggart (E.P. 263,275, 20.11.25).—Articles to be cleansed are immersed for a suitable period in a composition of sodium silicate (34 pts.), calcium carbonate (51 pts.), and water (12 pts.), maintained at about 77°. S. S. Woolf.

Making [aqueous] solutions of resins. Papeteries Navarre (E.P. 263,393, 28.5.26. Conv., 10.4.26).—Resin-bearing aqueous fluids, suitable for paper and card-board sizing etc., are obtained without the application of heat, by passing caustic soda solution, at a concentration lower than 10 g./litre, through vertical columns filled with the comminuted resin. S. S. Woolf.

Condensation products from *mono-* and di-cyclic phenols and aldehydes. A. Amann and E. Fonrobert, Assrs. to Chem. Fabr. Dr. K. Albert, G.m.b.H. (U.S.P. 1,614,171, 11.1.27. Appl., 13.12.23. Conv., 1.11.21).—Mono- or di-cyclic phenols in alkaline solution are added to aldehydes, the latter being in excess during the reaction. S. S. Woolf.

Condensation product of ketones and phenols. A. Amann and E. Fonrobert, Assrs. to Chem. Fabr. Dr. K. Albert, G.m.b.H. (U.S.P. 1,614,172, 11.1.27. Appl., 13.12.23. Conv., 25.4.21).—The hydroxyaryl-methane resulting from the condensation of a ketone with a phenol is treated with an aldehyde in an alkaline medium, at slightly above 60°. The resulting resin is readily soluble in dilute alkalis. S. S. Woolf.

Production of a neutral resin from gum accroides. K. H. T. Pfister, Assr. to Rohm & Haas Co. (U.S.P. 1,608,421, 23.11.26. Appl., 11.2.25).—Gum accroides (92 pts.) dissolved in alcohol (650 pts.) is treated with sodium hydroxide solution *d* 1·383 (38 pts.) and *p*-toluenesulphonyl chloride (106 pts.) at the b.p., when a tough, neutral resin, soluble in benzene, and insoluble in alcohol, is obtained. Other esterifying agents can also be employed. T. S. Wheeler.

Production of shellac substitutes. J. Scheiber and W. Noack (E.P. 252,715, 25.5.26. Conv., 29.5.25).—Shellac substitutes are obtained by condensing poly-hydroxy-fatty acids with hydroxycarboxylic acids of a hydro-aromatic nature (the analogues respectively of the aleuritic and shelloic acids composing the major part of natural shellac). In a typical example the mixed acids liberated from linseed oil and soft Manila resin are oxidised by permanganate, and heated together at 150° after purification. A clear resin is obtained, m.p. 90° (approx.), soluble in alcohol, alkali carbonates, and borax, but insoluble in fatty oils and benzene. S. S. Woolf.

Disposal of gases and vapours resulting from the boiling of oils, gums, resins, etc. D. E. Goggin (U.S.P. 1,612,915, 4.1.27. Appl., 29.12.24).—Varnish fumes etc. still sufficiently hot to preclude detrimental deposits of gummy substances are conveyed by a current of steam to a furnace in which they are burnt. S. S. Woolf.

Dispersing substances in water (U.S.P. 1,609,308).—See I.

Recovering resinous substances from cracked petroleum distillates (U.S.P. 1,608,135).—See II.

[Pigment from] reduction of aromatic nitro-compounds (E.P. 263,376).—See XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Revertex process. E. A. Hauser (Kautschuk, 1927, 2—16).—Revertex is obtained by the concentration of latex by evaporation together with an alkaline protective colloid in an externally heated rotating drum, inside which a freely rotating roller ensures thorough mixing. The pasty product, which contains only 20—30% of water, can be re-diluted for any purpose needing latex, and in this form can be vulcanised like ordinary latex. Vulcanised liquid latex also can be concentrated by the revertex process, and homogeneous mixtures suitable for the manufacture of "dipped" goods or unmasticated vulcanised sheet rubber can be obtained from it by incorporating fillers, preferably as colloidal suspensions. Compounding of revertex can conveniently be effected in a mechanical kneading machine into which the powders are introduced through a brush sieve, water also being introduced as necessary; the subsequent coagulation and drying may be effected on a pair of heated friction rolls, the final milling being completed on smooth rolls. If a content of carbon black is desired exceeding 20% (on the rubber) further quantities up to a total of 200% or more can be introduced during the drying operation. Revertex mixtures vulcanise more rapidly than ordinary rubber, and, as with most rubbers containing the whole of the serum solids, the vulcanised products "age" well. D. F. Twiss.

Rubber latex concentration and industrial application of concentrated latices. E. A. Hauser (Trans. Inst. Rubber Ind., 1926, **2**, 226—238).—A review of the subject with especial reference to the revertex process (see preceding abstract). D. F. Twiss.

Action of ultra-violet light on rubber solutions in the presence of yellow phosphorus. F. Kirchhof (Kautschuk, 1927, 28—30).—A benzene solution of rubber containing yellow phosphorus if exposed to ultra-violet light gels similarly to a rubber solution treated with dilute sulphur chloride. The dissolved yellow phosphorus is apparently converted into an ultramicroscopic dispersion of amorphous phosphorus. Zinc xanthate accelerates this "photo-gelation" of the mixture. The result is distinct from gelation by light alone, the resulting amorphous red phosphorus presumably exercising a "vulcanising" effect. D. F. Twiss.

***X*-Ray studies on rubber and similar substances.** E. A. Hauser and P. Rosbaud (Kautschuk, 1927, 17—21).—Calculation of the distance between the particles in unstretched crude rubber for the inner and outer amorphous rings gives results of 14·88 Å. and 6·05 Å., respectively. Metastyrene and a derived hydrocarbon $C_{16}H_{18}$ give diffraction patterns similar to those for unstretched raw rubber. D. F. Twiss.

What is rubber? E. A. Hauser (Trans. Inst. Rubber Ind., 1926, **2**, 239—244).—An account of chemical and physical views as to the structure of rubber. D. F. Twiss.

Fusion line of stretched rubber and its relation to the density. Law of elastic state. H. Feuchter (Kautschuk, 1927, 23—28).—Further consideration of the characteristics, particularly of the density and the rate of recovery when heated, of unvulcanised rubber which has been subjected repeatedly to a warming and stretching operation. The phenomena appear to accord with a "law of elastic state" in line with Le Chatelier's principle of "mobile equilibrium." The author's views as to the thermodynamics and structure of rubber are extended (cf. B., 1926, 204; 1927, 119). D. F. Twiss.

New hypothesis of rubber structure based on recent *X*-ray researches. E. A. Hauser (Ind. Eng. Chem., 1927, **19**, 169—170).—A re-statement of the view that the "crystalline" aggregates of highly poly-

merised rubber are normally swollen by the presence of the less polymerised portion of the hydrocarbon. When rubber is stressed, the swelling agent is expelled, and the amplitude of vibration of the crystalline aggregates is thereby reduced to such a degree that the possibility of definite X-ray interference results. D. F. Twiss.

Relation between vulcanisation by sulphur chloride vapour and the permanence of the vulcanised products. R. Ditmar (Chem.-Ztg., 1927, 51, 26—29).—An investigation of the effect of various conditions on the character of the vulcanised films of pale crêpe rubber, obtained with sulphur chloride, mainly by the "vapour cure." Careful adjustment of the degree of mastication of the rubber is necessary. Rubber solutions in benzene, with or without the addition of a small percentage of alcohol, yield transparent products of more attractive appearance than corresponding solutions in light petroleum. Atmospheric moisture leads to turbidity and loss of transparency. The inclusion of paraffin oil, soya bean oil, and, in particular, aniline in the rubber solution, has a favourable influence on the softness of the products; *Hevea* rubber seed oil is undesirable. Short immersion of the vulcanised film in a benzene solution of aldol-α-naphthylamine does not enhance its stability. A mixture of triphenyl phosphate and lycopodium, boric acid, and colloidal clay are satisfactory as dusting powders, but the last two somewhat impair the transparency. D. F. Twiss.

Selenium in rubber compounds. C. R. Boggs and E. M. Follansbee (Trans. Inst. Rubber Ind., 1926, **2**, 272—304).—A review of the uses of selenium in rubber compounding. Selenium can be used as a vulcanising agent or to accelerate vulcanisation by sulphur, the latter effect being attributed to the formation of selenium sulphide. The enhanced rigidity and abrasion resistance of tyre tread "compounds" vulcanised with sulphur and selenium in the presence of an organic accelerator are probably due to the joint addition of the two elements to the rubber molecule. D. F. Twiss.

Rubber softeners: their influence on ageing. W. N. Burbridge (Trans. Inst. Rubber Ind., 1926, **2**, 256—266. Cf. B., 1926, 598).—Features desirable from the use of a softener include an increase in the plasticity of the rubber with reduced resistance to milling, an enhanced capacity for absorption of compounding ingredients, and an improved resistance to ageing. Mineral oil, vaseline, ceresin wax, fatty oils, and fatty acids are classed as pseudo-softeners, and regarded as mere lubricants of the rubber cells, whilst naphthalene, mineral rubber, pine tar, rosin, and rosin oil are regarded as true softeners of the rubber nucleus. The true softeners, as a class, are more effective than the pseudo-softeners in their beneficial influence on the ageing properties of the mixing examined; exceptions in this respect include resin oil, oleic acid, and carnauba wax.
D. F. Twiss.

Effect of solid compounding ingredients as softeners [for rubber]. E. H. Hurlston (Trans. Inst. Rubber Ind., 1926, **2**, 267—271).—Not only can the plasticity of a rubber mixture be modified by the incorporation of soft wild rubbers or of definite "softeners," but many of the ordinary compounding ingredients have distinct influence. Organic accelerators, zinc oxide, red iron oxide, whiting, and gas black (in small proportion) exert a softening effect; barytes, lamp black, and china clay (in small proportions), lithopone, and antimony sulphide are neutral; magnesium carbonate and oxide, lime, litharge, and higher proportions of lamp black or gas black have a distinct "drying" influence.
D. F. Twiss.

Submarine insulation with special reference to the use of rubber. R. R. Williams and A. R. Kemp (J. Franklin Inst., 1927, **203**, 35—61).—The limited supply and high cost of gutta percha and balata necessitate a substitute for submarine insulation, and such a material must have mechanical adaptability, suitable specific electric constants, and permanence of mechanical and electrical characteristics, a common difficulty being excessive brittleness or lack of flexibility at sea-bottom temperature, and a high water absorption leading to an increase in dielectric constant. Rubber has adequate mechanical strength, but in its raw state is not highly plastic, and is too resilient, so that it cannot be extruded so easily as gutta percha. Soft vulcanised rubber, however, can be adapted to be mechanically and electrically suitable and to withstand the action of sea water in a manner comparable with gutta percha. The rubber insulation of the Seattle–Sitka cable is in an excellent state of preservation mechanically after sixteen years. The principal factor to be controlled is the amount of water absorption; gutta percha suffers some increase in dielectric constant from this cause, but rubber is still more affected. Porosity, the presence of proteins, and the osmotic pressure of internal and external fluids are important factors governing the inflow of water into rubber, but in oxygen-free salt solutions a definite limit of water absorption is reached. Lowered water absorption is attained by the removal of water-soluble matter from the rubber and by the use of suitable fillers. Fillers are required also to give the plasticity necessary to the good extrusion of rubber, but many are unsuitable because of high dielectric constant, or because of their solubility in water, or their tendency to form hydrates. They must be non-reactive and of suitable particle size, and have a minimum of adsorbed gases or other contamination on their surfaces. The best fillers for this purpose are hard rubber dust, silica roasted at 1000° for some hours to remove adsorbed gas, or zinc oxide.
W. G. Carey.

Yellow ink for marking rubber tensile strips. Cashion.—See XIII.

Patents.

Composition of matter [nitrated rubber]. H. L. Fisher, Assr. to B. F. Goodrich Co. (U.S.P. 1,609,806, 7.12.26. Appl., 19.10.22).—Rubber (500 g.), dissolved in carbon tetrachloride (6·25 litres), is agitated with concentrated nitric acid (500 c.c.) for 1 hr. The product (833 g.) has the probable formula $(C_5H_7NO_2)_x$, is soluble in acetone and nitrobenzene, insoluble in benzene, and is useful in the preparation of insulating materials. It decomposes at about 136°, and, when treated with methyl sulphate, yields a substance stable above 300°.
T. S. Wheeler.

Composition prepared from bitumen and rubber. J. Campbell (E.P. 263,028, 29.5.26).—A material suitable

as a dressing for roads, as a protection for iron, steel, or wood, etc., is produced by stirring a mixture of ground scrap vulcanised rubber (70 pts.) with molten asphaltum or petroleum residue (30 pts.) until homogeneous. The characteristics of the mixture may be modified by the inclusion of a small proportion of pitch, resin, sulphur, rubber solvent or solution of rubber, fibrous material, and plaster of Paris. D. F. TWISS.

Vulcanisation of caoutchouc. C. W. BEDFORD, Assr. to B. F. GOODRICH Co. and GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,613,572—3, 4.1.27. Appl., [A], 28.12.23. Renewed 5.6.26. [B] 11.2.25).—(A) Rubber containing an organic disulphide having a double linking adjacent to the C·S·S·C group is treated with hydrogen sulphide to produce the C·SH group, and is then vulcanised. (B) Vulcanisation of rubber is accelerated by successive treatment with a plurality of substances which combine together to form a dithiocarbamic compound. D. F. TWISS.

Accelerators for rubber vulcanisation. E. C. R. MARKS. From E. I. DU PONT DE NEMOURS & Co. (E.P. 263,517, 17.9.25).—The vulcanisation accelerators produced by the condensation of aromatic amines with an aldehyde containing a plurality of carbon atoms are commonly sticky, viscous fluids. By further heating, *e.g.*, between 80° and 100°, with formaldehyde they are converted into paler, brittle, solid products which are more convenient for use. (Reference is directed, in pursuance of Sect. 8, Sub-sect. 2, of the Patents and Designs Acts, 1907 and 1919, to E.P. 251,005. See B., 1926, 555.) D. F. TWISS.

Device for registering the degrees of hardness of vulcanised rubber or other resilient materials. DUNLOP RUBBER Co., and E. SIMPSON (E.P. 263,636, 18.1.26).—An instrument capable of measuring small differences in hardness of resilient materials is operated by pressing a projecting plunger against the article under test. The spring maintaining the plunger in position is so selected that the latter penetrates considerably before the spring is depressed. The full range of the scale reading therefore corresponds with a limited range of variation in hardness. For convenience in reading, a device is included enabling the fixing of the dial needle. D. F. TWISS.

XV.—LEATHER; GLUE.

***X*-Ray spectrographic examination of the tanning of membranes and tendons.** J. R. KATZ and O. GERNGROSS (Kolloid-Z., 1926, 40, 332—333).—The *X*-ray spectrograph obtained from untanned leather consists of a broad inner ring characteristic of the amorphous state, with an outer ring typical of interference due to crystalline arrangement. Various specimens of leather differ only in the intensity of these rings. With threads of collagen, tanning with formaldehyde does not alter the *X*-ray interference figure, a difference being noticed only when an extract of gall-nuts is used as the tanning agent. It is supposed that only the outermost layer of molecules of collagen is affected. E. S. HEDGES.

Fluorescence of wood cellulose and vegetable tanning extracts. GERNGROSS.—See V.

PATENTS.

Production of gelatin and glue from chrome-leather or its shavings. J. RINCK (U.S.P. 1,612,746, 28.12.26. Appl., 4.9.24. Conv., 28.1.24).—See E.P. 226,722; B., 1924, 140.

Manufacture of artificial leather. P. MAGNUS (E.P. 263,004, 22.3.26).

XVI.—AGRICULTURE.

Sulphuric acid treatment of cotton seed. R. G. ARCHIBALD (Soil Sci., 1927, 23, 1—3).—Cotton seeds (500 g.) were treated with concentrated sulphuric acid (100 c.c.) for 15 min., washed for 10 min. in 2 litres of water, and dried. Sown immediately after treatment, the seed showed 95% germination, and, after storage for 6 months, 92%. In the field treated seed germinated better than the untreated. Soaking the seeds in acid for longer periods than one hour, even with a dilution of 1 : 500, affected their germinative power. Seeds infected with "Black Arm" were not completely sterilised by the acid treatment, but the active development of the disease was delayed, and young plants became well established before any injury was apparent. A. G. POLLARD.

Use of collodion sacs in obtaining clear soil extracts for the determination of the water-soluble constituents. W. H. PIERRE and F. W. PARKER (Soil Sci., 1927, 23, 13—32).—The difficulty of filtering soil extracts without loss of calcium and phosphate is overcome by the use of collodion sacs. Prepared as described, the sacs are without effect on soluble ions. Equilibrium between soil suspension and dialysate is reached within 24 hrs., and with the use of a mechanical shaker in a much shorter period. Hydrogen-ion concentrations in dialysed soil extracts agree well with values determined electrically. The use of collodion sacs in studies of the buffer capacity of soils, the soluble mineral matter, etc. is indicated. A. G. POLLARD.

Occurrence of yeast in soil. R. L. STARKEY and A. T. HENRICI (Soil Sci., 1927, 23, 33—45).—Various species of yeasts were found in small numbers in many soils examined. The presence of yeasts was in no way dependent on the type of soil, the crop, or season. A. G. POLLARD.

Obligate thermophilic bacteria from soil. W. A. FEIRER (Soil Sci., 1927, 23, 47—56).—A number of species of aerobic, obligate, thermophilic bacteria were isolated from surface soils. Their characteristics are described. Optimum growth occurred in the range 50—60°, and the death-point range was 125—155°. Organisms were not pathogenic to guinea-pigs. One species exhibited proteolytic activity. A. G. POLLARD.

Oxidation of sulphur by micro-organisms during ammonification. G. GUITTONNEAU (Compt. rend., 1927, 184, 45—46. Cf. A., 1925, i, 1218).—Certain ammonising micro-organisms were placed in solutions containing peptone and free sulphur, and after 40—50 days it was found that in most cases thiosulphates had been formed. When the pure cultures were aided by the action of bacterium b_2 (B., 1925, 774), further oxidation to sulphates occurred. *Aspergillus niger* and *Penicillium glaucum* caused oxidation to sulphates without

the aid of b_2. The presence of sulphur thus affects the equilibrium during ammonification. B. W. Anderson.

Agrological investigations of the dynamics of biochemical processes in podsol soils. S. P. Kravkov (Pochvovedenie [Russia], 1925, **20**, 5—19).—Determinations of nitrates, ammonia, total solids, organic and mineral matter, reaction, phosphorus, calcium, potassium, etc. in aqueous extracts of soils indicate that despite variations in meteorological and other conditions the type of curve of nitrate formation (and apparently of total solids) in natural soils remains the same. A. A. Eldridge.

Sunlight and chemical nitrification. I. P. Zholtzinskii (Pochvovedenie [Russia], 1924, **19**, 80—97).—Cellulose was treated with hot sulphuric acid (*d* 1·84, 100 c.c.) and water (50 c.c.), washed, dialysed, and saturated with ammonia (1·83%) for 8 days. The dark-coloured filtrate, containing 0·48 g. of humus substances per litre, on exposure to sunlight became light in colour, and gave reactions for nitrates, the humic substances catalysing the oxidation. Experiments with 1—2% solutions of substances of the benzene structure showed that in photochemical humification hydrogen peroxide is produced.
A. A. Eldridge.

Soil structure and its significance to agriculture. K. K. Gedroiz (Ann. Inst. Exp. Agron. [Russia], 1926, **4**, 117—127).—Soil structure is determined by pressure (*e.g.*, of clover roots) and coagulation. The coagulating power of cations is in the increasing order: lithium, ammonium, potassium, magnesium, calcium, hydrogen, aluminium, iron. Hydroxyl ions hinder coagulation, but the stabilising action of other anions is insufficient to offset the effect of the cations. Coagulation is closely connected with replacement and adsorption in the colloidal complex capable of base exchange.
A. A. Eldridge.

Origin of alkali soils. D. G. Vilenskii (Pochvovedenie [Russia], 1924, **19**, 36—58).—Alkali soils are formed from salinised soils when the latter lose their contact with the ground waters. A. A. Eldridge.

Colloidal fungicides. J. Bordas (Rev. gén. Colloid., 1926, **4**, 289—294).—The advantages of using fungicides in a colloidal state, both for spraying and seed disinfection, are discussed, and a short account is given of a number of such preparations, the most important of which contain either sulphur, copper compounds, or mercury sulphide. Some recent practical experiments, which have given promising results, are briefly noted.
C. T. Gimingham.

Cellulose [bacteria]. I. A. Itano (Ber. Ohara Inst. landw. Forsch., 1926, **3**, 215—219).—A preliminary note, dealing mainly with the cellulose-decomposing powers of an aerobic, thermophilic bacterium. The organism is active both in pure culture and in composts of vegetable waste materials. C. T. Gimingham.

Applicability of the indirect method of analysis to determination of sodium and potassium in soil solutions. R. E. Neidig and W. B. Bollen (Ind. Eng. Chem., 1927, **19**, 154—156).—The formulæ used are essentially those of Crookes except that the factors are based on at. wts. from the International Tables. Indirect determination of sodium and potassium in solutions of their chlorides showed some individual errors even exceeding 2 mg., but the average was less than 1 mg. for Na : K or K : Na ratios of 0 : 100. In comparison with the chloroplatinate method of determination for soil extracts, the indirect method from a determination of the chloride is regarded as equal in accuracy, except where potassium alone is sought, and is superior from the standpoint of economy of time and cost of reagents. D. G. Hewer.

Storage of rice and changes of its physical properties during this period. M. Kondō (Ber. Ohara Inst. landw. Forsch., 1926, **3**, 153—175).—The variations in temperature of stored hulled rice, in relation to the air temperature, have been studied, and observations made on the changes in water content, water-absorbing capacity, hardness, and other properties of the rice. Practical conclusions are drawn as to suitable conditions for the storage of hulled rice. C. T. Gimingham.

Biilmann's quinhydrone electrode. I. Table for p_H values corresponding to electromotive forces determined in quinhydrone electrode measurements. II. A. Itano and K. Hosoda (Ber. Ohara Inst. landw. Forsch., 1926, **3**, 193—201, 203—214).—A number of points in connexion with the working of the quinhydrone electrode and the arrangement of the apparatus are discussed. The method is considered to have advantages over the use of the hydrogen electrode, particularly for soil investigations. A table is given of p_H values corresponding to the *E.M.F.* measured by means of the quinhydrone electrode.
C. T. Gimingham.

Soil micro-organisms and activators. A. Itano (Ber. Ohara Inst. landw. Forsch., 1926, **3**, 185—191).

Extraction of copper ores with ammonia and production of arsenic-copper preparations. Fester and Bertuzzi.—See X.

Patents.

Manufacture of superphosphate. B. Bodrero (E.P. 262,878, 22.9.25).—The raw phosphate is subjected to a preliminary washing with water in order to remove some calcium carbonate and render the material more porous. The material is then mixed with 3·5—10% of sulphur and, in some cases, putrefiable organic matter, and maintained at 40—50° in silos or ovens, in which it receives injections of water vapour. Under these conditions sulphur dioxide is produced and reacts with the mineral phosphate. G. W. Robinson.

Manufacture of phosphate fertiliser. L. Adelantado (E.P. 262,833, 9.6.25).—In the manufacture of mixed fertilisers containing water-soluble phosphates of the type M'_2HPO_4 and phosphates soluble in citric acid of the type $CaM'PO_4$, iron and aluminium phosphates or natural phosphates containing substantial amounts of iron and aluminium are treated with sulphates of the alkali metals or magnesium together with calcium carbonate, and a small quantity of sulphuric acid in the presence of water, and without the application of heat. The amount of acid used is equivalent to that required to convert all the phosphate, expressed as tricalcium phosphate, into the dicalcium phosphate. By

increasing the speed of reaction, working at a low temperature, using a slight excess of acid, and storing so that heat is rapidly dissipated, a product rich in water-soluble phosphate is formed. By varying the conditions a product low in water-soluble phosphate but rich in citric-soluble phosphate may be obtained. Organic matter, *e.g.*, peat or sewage sludge, may be added if desired. G. W. ROBINSON.

Dry fertilisers and glycerin from vinasses. E. A. BARBET (E.P. 263,322, 30.1.26).—See F.P. 605,825; B., 1926, 893.

XVII.—SUGARS; STARCHES; GUMS.

Composition of mesquite gum: isolation of *d*-galactose and *l*-arabinose. E. ANDERSON and L. SANDS (J. Amer. Chem. Soc., 1926, **48**, 3172—3177).—Hydrolysis of mesquite gum with 4% sulphuric acid at 80° affords *l*-arabinose (50·7%), which was separated from the calcium salts obtained by neutralising the solution with calcium carbonate. Further hydrolysis of these salts by 3% sulphuric acid in an autoclave at 1 atm. gauge pressure yields *d*-galactose (18·7%), and an *aldehydic acid* of the galacturonic acid group (13%). These three constituents, together with moisture, ash, and a small proportion of nitrogenous material, are practically the sole constituents of the gum. F. G. WILLSON.

Preparation of sugar from sawdust. HÄGGLUND.—See V.

XVIII.—FERMENTATION INDUSTRIES.

Experimental brewings with new varieties of hops. J. M. LONES and T. E. GRANT (J. Inst. Brew., 1927, **33**, 6—11).—Three new varieties of hops which showed exceptionally high percentages of resins for English-grown hops were put on trial both as copper hops and as racking hops, and were also compared with a good quality British Columbian, 1924, crop. The antiseptic values determined chemically were 84, 54, and 76, and by the Chapman biological method 63, 83, and 90. The British Columbian hop, by the latter method, gave a value of 70. In brewing, fermentation, and racking no noticeable difference was observed between any of the brewings, whilst the weight, quality, and purity of the yeast outcrops showed very little variation. Compared with the British Columbian, the new varieties gave rise to slightly lower attenuations, and had equal flavouring properties, but possessed a somewhat inferior brewing value so far as keeping properties were concerned. With the exception of the foreign hop, the keeping properties showed a rough approximation to the preservative values as determined by the Chapman method. The "brewing value" appeared to follow very closely the "rub" of the hops. C. RANKEN.

Determination of amylase in the mash. F. WENDEL (Z. Spiritusind., 1927, **50**, 9).—To determine whether the necessary excess of amylase is present in distillery wort, 5 c.c. of clear wort are added to 15 c.c. of a 1% solution of soluble starch. The mixture is incubated at 27—28° for 1 hr. and a few drops of iodine solution are added. According as the resulting colour is yellow, reddish, or bluish-violet, an excess, sufficiency, or deficiency of amylase is present. To avoid the need for an accurate balance and the preparation of large quantities of soluble starch solution, the soluble starch should be purchased in its prepared state in glass tubes each containing 2 g. C. RANKEN.

Wines from vintages attacked by the *Cochylis* and *Eudemis*. L. FERRÉ (Ann. Falsif., 1926, **19**, 595—599).—Wines made from grapes attacked by the *Lepidoptera Cochylis* and *Eudemis* show chemical differences from the normal depending partly on the locality, but chiefly on the time of attack, so that natural chemical changes in the grape may be arrested at different stages. The dry extract is increased by 10—50% above the average. Total acidity differs considerably according to whether litmus or phenolphthalein is used as indicator, owing to the presence of weak organic acids of high mol. wt. The proportion of malic and tartaric acids depends largely on the time when development was arrested; alcohol precipitates substances convertible into sugars by 2% hydrochloric acid at 120°, probably of the nature of gums and pectic substances; the proportion of glycerin is increased, and the ratio of alcohol to glycerin, which is always greater than 10 in normal Burgundy wines, falls below 10. Tannic material, mineral and nitrogenous substances are present in greater proportions than normal. D. G. HEWER.

Butyl alcohol and acetone from corn [maize]. D. H. KILLEFFER (Ind. Eng. Chem., 1927, **19**, 46—50).—Butyl alcohol, acetone, and ethyl alcohol are produced in the ratio of 6 : 3 : 1 by fermentation of low-grade maize by means of cultures of *Clostridium acetobutylicum*. This culture converts 3 lb. of starch into 1 lb. of mixed solvents in the form of a 2·5% solution, and a mixture containing 45% H and 55% CO_2 is evolved as a by-product. The solvent mixture is passed through continuous stills, and the resulting crude spirit (50% solvents) then fractionated. D. G. HEWER.

Purification of waste water from the fermentation industries. G. BODE (Z. angew. Chem., 1927, **40**, 84—87).—The discharge of objectionable waste water from breweries is prevented by purifying the highly-contaminated water from the brewing house and the cellars before it is discharged into the main sewer and contaminates the remainder, which is comparatively innocuous. The impurities, being mainly of a solid nature, such as coagulated albuminous material, hop resins, and yeast, can be removed by sedimentation, and apparatus for this purpose is described. In potato distilleries, the residues are worked up into cattle fodder, and the waste water is not contaminated. The purification of the water from distilleries using molasses, the still residues of which contain much putrescible organic material, and of waste liquors from the manufacture of compressed yeast from molasses requires further attention. L. A. COLES.

Oxidation of sulphur by micro-organisms during ammonification. GUITTONNEAU.—See XVI.

Absorption by vegetable tissues. EFFRONT.—See XIX.

PATENTS.

Extraction of tartaric [acid] salts [from wine-lees etc.]. W. E. KLAVERSTEIJN (E.P. 263,340, 15.2.26).

—The residues of wine-making (lees, tartars, or argols, etc.) are dried, ground, and roasted at 150°. Water or mother-liquor from a previous operation is added, and the liquid saturated with sulphur dioxide, which is maintained at a pressure of 3 atm. for 30 min. The potassium hydrogen tartrate and calcium tartrate of the wine residues are converted into soluble tartaric acid and the metal bisulphites. The liquid is filtered and the pressure reduced, when the above reaction is reversed, potassium hydrogen tartrate being precipitated. The reaction is interrupted when 2% of the latter remains, in order to prevent the calcium tartrate from precipitating. The mother-liquor is used again, being finally evaporated for the production of calcium tartrate.
B. Fullman.

Fertiliser and glycerin from vinasses (E.P. 263,322).—See XVI.

Production of leavened bread and other dough products (E.P. 235,874).—See XIX.

XIX.—FOODS.

Xylene and xylene percentage numbers [in examination of butter]. A. van Raalte (Chem. Weekblad, 1927, **24**, 59—60. Cf. B., 1926, 563).—The formula previously given is modified for percentage of milk fat to (xylene number $-0\cdot63) \div 0\cdot20$; the value 0·63 is not constant, but varies with the amounts of cacao butter and/or palm-kernel oil present. From examination of 108 samples, it is concluded that a butter having a lower xylene percentage number than 66 must be regarded as adulterated. S. I. Levy.

Examination of sterilised milk 27 years old. G. Schulze (Z. Unters. Lebensm., 1926, **52**, 380—384).—Apart from a separation into three distinct layers very little change had occurred in the milk. The upper layer resembled in composition and properties a fresh butter fat. In the middle watery layer the normal relationship between the lactose and the chloride persisted, and although the casein was somewhat less, the albumin and the residual nitrogen fell within the usual limits. Part of the lime and phosphoric acid of the milk had separated out as an insoluble crystalline phosphate, together with tyrosine, at the bottom of the container. F. R. Ennos.

Power of absorption of vegetable tissues. J. Effront (Chim. et Ind., 1926, **16**, 376—384, 556—560, 730—736, 908—912).—A general account of absorption phenomena in which vegetable and animal tissues take part. The power of absorbing water possessed by wood varies greatly with the species, whilst that of fruit pulp is shown to vary with the hydrogen-ion concentration. The physical properties of yeast depend largely on the ratio between the amount of water absorbed by the cells and that held mechanically in the interstices. The consistency of the yeast varies with the amount of adhering water, which can be increased by the addition of salts, such as ammonium sulphate. The activity of the yeast is unaffected by this treatment. The absorbing power of fruit pulp for pepsin decreases as the fruit approaches maturity, but for acid or alkali the variation is slight. The ash content of the fruit falls as ripening progresses, hence the decrease in the pepsin-absorbing power. Fruit after boiling with water for 5—6 hrs. is capable of absorbing an increased quantity of $0\cdot1N$-hydrochloric acid, but its affinity for $0\cdot1N$-sodium hydroxide is reduced. Drying the pulp at 80—100° reduces the absorbing power for both acid and alkali. The amount of pepsin absorbed by cellulose varies greatly with different samples, and depends rather on the physical structure of the latter than on their chemical purity. The proteins present as impurities in the pepsin are not absorbed, and by this method a very active sample of pepsin containing only 0·4% N can be obtained. Vegetable juices which have lost their hydrolysing power by being heated can be reactivated by simple filtration through paper, which absorbs the retarding substances formed during the heat treatment. The theoretical principles underlying absorption phenomena in therapeutics and in normal nutrition are discussed. W. J. Powell.

Determination of caffeine. S. Gobert (Ann. Falsif., 1926, **19**, 586—594).—The following method gave satisfactory results for the determination of caffeine both in roasted and green coffee, and except in de-caffeinised samples the determination of nitrogen in the extracted residue is unnecessary. To 5 g. of finely-ground coffee 5 c.c. of ammonia solution (*d* 1·18) are added and left in contact with roasted coffee for 20 min. or with green coffee 30 min., with occasional shaking. Four extractions with 25 c.c. of ethyl acetate are then made, contact being in each case for 10 min., with continual shaking. After centrifuging for 5—7 min., decanting the liquor, and adding 0·5 g. of paraffin, the ethyl acetate is distilled off, and the residue dried, extracted three times with 50 c.c. of boiling water, and the united extracts are boiled, cooled, and filtered, and 20 c.c. of 1% potassium permanganate solution are added for roasted, and 10 c.c. for green coffee, and left for ¼ hr. The manganese is then precipitated by adding 12 vol. hydrogen peroxide containing 1% of glacial acetic acid and leaving for ¼ hr. in the water bath. The liquid is filtered, the precipitate washed with boiling water, and the filtrate evaporated, dried, and extracted with warm chloroform, filtered, and washed with 15 c.c. of chloroform. The chloroform is then distilled off and the residue dried and weighed. Moisture should be determined at the same time as the caffeine.
D. G. Hewer.

Caffeine content of coffee extracts and their physiological action. H. Jesser (Z. Unters. Lebensm., 1926, **52**, 389—392).—The caffeine contents of extracts made from roasted coffee with equal vols. of water and of a solution of sugar in water, are practically identical. The statement that coffee made by adding sugar before extraction has less action on the heart than that in which the sugar is added afterwards is not confirmed.
F. R. Ennos.

Determination of caffeine in black tea. W. Stüber (Z. Unters. Lebensm., 1926, **52**, 393—395).—5 g. of powdered tea are treated with 5 g. of 10% ammonia solution and 200 g. of chloroform and shaken for ½ hr. The whole is filtered, 150 c.c. of the filtrate are evaporated to dryness, and the residue is dissolved in 80 c.c of hot water. 10 c.c. of 1% potassium permanganate solution are added to the cooled

solution, which is boiled for $\frac{1}{4}$ hr., the excess being reduced by the addition of hydrogen peroxide acidified with acetic acid. After filtering, the solution is extracted first with 50 c.c. of chloroform, followed by three further extractions with 25 c.c. each. The combined extracts are evaporated to dryness, and the caffeine residue is dried to constant weight at 100°. F. R. ENNOS.

Detection of hydrogen peroxide in beverages preserved with this compound. C. D. HOWARD and N. CIVEN (Ind. Eng. Chem., 1927, **19**, 161—162).—Hydrogen peroxide used to preserve beverages does not decompose with such rapidity as has been sometimes assumed. The starch–potassium iodide test has been found reliable for the detection of small quantities of hydrogen perixode. 1—2 c.c. of the reagent are added to 10 c.c. of fresh milk and about 5 c.c. of the sample, either by flowing on top for the production of a blue zone or by mixing to give the uniform blue colour due to small amounts of hydrogen peroxide. If benzidine or *p*-phenylenediamine is used, slight positive tests may be given on controls by the zone method, and no blue colour on mixing. The vanadic acid test is worthless, as controls of cocoa and sugar alone give a positive test. D. G. HEWER.

Nutrose and eucasin. M. A. RAKUSIN and K. BRAUDO (Z. Unters. Lebensm., 1926, **52**, 396—397).—Commercial nutrose is shown to be identical with sodium caseinate prepared by neutralisation of casein with sodium hydroxide and filtration through a Pukalk–Berkefeld filter. The eucasin of commerce, which is ammonium caseinate, is obtained by dissolving casein in ammonia and removing the excess of the latter by evaporation. It does not give the Ostromislenski reaction. From a nutrose solution caseinates of the heavy metals are obtained as coloured substances by double decomposition with their salts. F. R. ENNOS.

Chondrin and gluten. M. A. RAKUSIN and K. BRAUDO (Z. Unters. Lebensm., 1926, **52**, 397—401. Cf. B., 1923, 851).—The chondrin molecule is dissociated in dilute solution, and on treatment of the opalescent (0·2%) solution with aluminium hydroxide part is absorbed leaving a clear solution of chondroitinsulphuric acid. With a 0·5% solution the biuret complex only is broken up, and with a still stronger solution (1·2%) a true absorption of the chondrin molecule by the alumina takes place. By evaporation of the clear solution the acid may be obtained as a crystalline powder (decomp. 180°). It has a strong negative rotation, shows none of the protein reactions, and gives a precipitate with barium chloride. In the case of clear gelatin solutions the detection and determination of this acid may be carried out by means of barium chloride, without any preliminary treatment with aluminium hydroxide. F. R. ENNOS.

Chemical constituents which influence gluten quality. B. SULLIVAN and C. NEAR (Ind. Eng. Chem., 1927, **19**, 159—161).—Gluten prepared from straight flours milled in a small experimental mill from strong Marquis wheat, and from patent and clear flours by a long milling process from the same wheat, were analysed, and the totals of starch, lipoids, protein, and ash in each case were slightly over 100%. The best quality gluten (the patent) had the lowest lipoid content. Analyses of the ashes showed that the proportion of calcium decreased with the quality of the gluten, but potassium and magnesium varied in the opposite direction. Lipoids were determined by refluxing a mixture of the ground dry gluten (3 g.) with twice its weight of fine pumice in an alundum extraction cylinder of suitable porosity with a mixture of 90 c.c. of 95% alcohol, 5 c.c. of concentrated ammonia, and 5 c.c. of water, followed by extraction with ethyl ether for 2—3 hrs. The mixture of the alcohol and ether extracts is then evaporated, the residue taken up with chloroform or carbon tetrachloride, filtered if necessary, evaporated, and the residue dried to constant weight. D. G. HEWER.

Determination of cacao shell by sedimentation. J. GROSSFELD (Z. Unters. Lebensm., 1926, **52**, 343—360 Cf. B., 1926, 688).—Several formulæ are developed for calculating the amount of cacao shell from the percentage of sedimentation residue and its fibre content. Experiments with mixtures of known shell content confirm the results obtained by means of these formulæ, provided the percentage of shell is not very small. By repeated sedimentation on the first residue after air-drying and grinding, as little as 0·5% of shell can be detected. Mixtures containing shell powder and cacao germ give low figures for the shell content. The presence of cacao germ is indicated by an increase in shell content found after grinding the first sedimentation residue, and repeating the sedimentation several times. Neither the phosphoric acid content of the residue, obtained by ordinary sedimentation or by sedimentation in the presence of soluble calcium salts, nor the amount of carbohydrates obtained from the residue by hydrolysis, forms a satisfactory basis for calculating the shell content of a cacao product. F. R. ENNOS.

Examination of cacao beans and cacao products. IV. H. FINCKE (Z. Unters. Lebensm., 1926, **52**, 360—364. Cf. B., 1924, 489; 1925, 50; 1926, 73).—Cacao shell in its natural state has a small amount, usually less than 1% and never more than 2%, of a characteristic, viscous, yellowish-brown fat of high acidity. It is free from theobromine. Cacao shell separated from the commercial beans often has a higher percentage of fat and shows the presence of theobromine, due probably to the introduction of these constituents during the fermentation process. Cacao butter shows on the average 0·006% of lime-bearing ash. F. R. ENNOS.

"Cryolac" number of milk and milk products as a means to determine quantity of added water. P. POST (Z. Unters. Lebensm., 1926, **52**, 371—380).—Cf. B., 1926, 846.

New index for butter fat. MORGENSTERN.—See XII.

Storage of rice and changes of its physical properties during this period. KONDŌ.—See XVI.

PATENTS.

Production of leavened bread and other dough products. T. B. WAGNER and C. A. GLABAU (E.P. 235,874, 13.6.25. Conv., 21.6.24).—The time required for leavening is considerably reduced by mixing the flour, yeast, and other ingredients with a yeast activator, which is preferably prepared from "corn solubles" and contains a large amount of phosphorus and calcium in organic combination. A gluten maturing agent, such

as lactic, hydrochloric, or other acids, is then added in sufficient quantity to form with the water in the mixture a solution strong enough to mature the gluten in 1 hr., after which period the dough is baked. F. R. Ennos.

Cultured-milk drink. T. H. Mittendorf (U.S.P. 1,610,962, 14.12.26. Appl., 19.2.26).—Selected strains of bacteria are introduced into milk, which is cooled to 3°, and agitated *in vacuo*. It is then treated with carbon dioxide for several hrs. at a pressure such that the cultures are not destroyed, but reduced to a state of suspended animation, whence they may regain their biochemical activity by the restoration of atmospheric pressure. The pressure is then reduced, but not removed, and the milk bottled. F. R. Ennos.

Treatment of nut kernels. J. A. Thompson and W. S. Hough (E.P. 263,518, 18.9.25).—Brazil nut kernels are blanched by subjecting them, while free to move in a closed vessel of smooth internal contour, for about 15 sec. to a blast of steam superheated to 200°, which removes the skins without cooking the kernels. F. R. Ennos.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OIL.

Action of hydrogen chloride on methyl alcohol. S. R. Carter and N. J. L. Megson (J.S.C.I., 1927, **49**, 31—35 T).—The investigation of the interaction of hydrogen chloride and methyl alcohol (Carter and Butler, A., 1924, i, 603) has been extended, and it is found that in the gaseous state there is little or no tendency to combine over periods up to 45 hrs. at 100°, 150°, and 200° respectively, provided that all condensation is rigorously prevented. At 60°, at which partial condensation may occur, there is appreciable combination between methyl alcohol and hydrogen chloride. This tends to the same limiting value (57% decrease of hydrogen chloride in 87 hrs.) as the solution does at room temperature (57% decrease after 134 days). Methyl alcohol and hydrogen chloride react in the liquid state under conditions which would be expected from the law of mass action. In closed vessels the methyl chloride produced causes an excess pressure which tends to retard the reaction. In open vessels a retardation is also observed, on account of the evaporation of methyl alcohol, and consequent concentration of the hydrogen chloride. The inhibiting effect of water on the reaction in the liquid state is also in accordance with the law of mass action.

Analysis of mixtures containing water, alcohol, and ether. Chenel (Mém. Poudres, 1926, **22**, 145—147).—Air freed from moisture and carbon dioxide is passed at the rate of 300—500 c.c./hr. over the mixture to be analysed, and then through three U-tubes containing potassium carbonate, manganous chloride, and pumice soaked in sulphuric acid respectively. These tubes in turn retain water, alcohol, and ether, which may be determined quantitatively by finding the increase in weight of the tubes. By aspirating air for 22 hrs. over 2·022 g. of a mixture containing water 13·1%, alcohol 26·1%, and ether 60·8%, the values thus obtained were 13·5, 25·8, and 60·7. S. Binning.

Determination of mercury in mercuric salicylate. A. F. Murray (Amer. J. Pharm., 1926, **98**, 639—642).—The following is a shorter and more accurate method than that given in the U.S.P. for the assay of mercury in mercuric salicylate. Dissolve 0·5 g. of mercuric salicylate in 10 c.c. of 10% aqueous sodium hydroxide, with warming. Add 10 c.c. of 10% sodium sulphide solution and boil. Acidify the hot solution with 10% hydrochloric acid, and add 5 c.c. excess. Filter hot through a prepared Gooch crucible, dried at 110°, and wash with hot distilled water until free from chlorides. Wash twice with 5 c.c. of alcohol, then with 5 c.c. portions of equal parts of ether and alcohol until the washings give no reaction with ferric chloride. Finally, wash with three 5 c.c. portions of carbon tetrachloride, dry at 110°, and weigh. B. W. Anderson.

Micrographic detection of tartaric acid in official preparations. M. François and C. Lormand (Ann. Falsif., 1926, **19**, 599—605. Cf. B., 1926, 802).—Tartaric acid may be detected by the characteristic microscopical appearance of the crystals of the *d*-acid as follows:—For syrups and lemonades 100 c.c. are diluted with 80 c.c. of water, filtered, and 20 c.c. of calcium acetate solution added, and after 3 days the crystals formed are filtered off, washed with alcohol at 32°, dried in the air, and examined. The test may be confirmed by means of sulphuric acid and resorcinol. In the case of syrup of iron iodide, the iron must first be eliminated. In wines, elixirs, and medicinal vinegars most of the sulphates present must first be eliminated to obtain pure crystals, and 25 c.c. of normal lead acetate solution (20%) are added to 100 c.c. of the sample, followed by 25 c.c. of sodium carbonate crystals (40%). The liquid is filtered after 15 min., made up to 180 c.c., 4 c.c. of glacial acetic acid are added, and 20 c.c. of calcium acetate solution. After 3 days the deposit is examined microscopically. For some wines the solubility of the calcium tartrate must be diminished by the addition of alcohol. For saline compounds, *e.g.*, Sedlitz water, magnesium sulphate must be removed by addition of barium chloride solution, followed by sodium carbonate, to precipitate excess of barium and magnesium. After filtration, the filtrate is acidified with glacial acetic acid, and calcium acetate solution is added, when the crystals form slowly. D. G. Hewer.

Synthetic drugs. I. Significance of the acyl group in acylated drugs. H. P. Kaufmann (Z. angew. Chem., 1927, **40**, 69—79).—In order to extend the knowledge of the effect of the addition of acyl groups upon the physiological action of drugs, a number of new compounds have been prepared, including: *octoylsalicylic acid*, m.p. 70°; *decoylsalicylic acid*, m.p. 81°; *laurylsalicylic acid*, m.p. 77·5°; *myristylsalicylic acid*, m.p. 70°; *palmitylsalicylic acid*, m.p. 73—74°; *stearylsalicylic acid*, m.p. 88°; o-*phthalyl*-p-*phenetidine*, m.p. 189°; o-*phthalylhydrazine*, m.p. > 315°; o-*phthalylphenylmethylhydrazine*, m.p. 162°; o-*phthalyldiacetylphenylhydrazine*, m.p. 247°; o-*phthalyldiformylphenylhydrazine*, m.p. 214°; o-*phthalylhydrazobenzene*, m.p. 174°; o-*phthalylhydrazotoluene*, m.p. 174°; *succinylhydrazobenzene*, m.p. 245°; *dibenzyl*-o-*phthalylhydrazine*, m.p. 137°; 1-*phenyl*-2-*allyl*-4-*diethyl*-3 : 5-*diketopyrazolidine*, b.p. 190—200°/19 mm.; 1 : 2-*dibenzyl*-4-*diethyl*-3 : 5-*diketopyrazolidine*, yellow oil; 2-*acetyl*-

1-*phenyl-4-diethyl-3* : 5-*diketopyrazolidine*, m.p. 96°; and 2-*formyl-1-phenyl-4-diethyl-3* : 5-*diketopyrazolidine*, m.p. 110°. In the acylated salicylic acid series, resistance to hydrolysis by dilute alkalis, *i.e.*, under the conditions prevailing in the intestines, increases with increasing mol. wt. When the members of the series are taken internally, salicylic acid can be detected in the urine sooner, but persists for a shorter time the lower the mol. wt. The partition coefficient, $C_{\text{olive oil}}/C_{\text{water}}$, which is of physiological importance in indicating the extent to which the compounds can be absorbed by the lipoids, is 5·1 for salicylic acid, 1·17 for acetylsalicylic acid, and for decoylsalicylic acid and higher members is ∞. Dilute salicylic acid solutions (0·01—0·0033*N*) had no narcotic effect on leeches, but caused death by irritation of the mucous membrane, whereas acetylsalicylic acid solutions caused marked narcosis, and were only toxic at the highest concentration (0·01*N*). Sodium salicylate is less toxic than the free acid, but causes no narcosis. The narcotic effect of sodium decoylsalicylate is less marked than that of sodium acetylsalicylate. L. A. COLES.

Analysis of the fluid extract of *Hydrastis Canadensis*. F. E. RAURICH (Anal. Fís. Quím., 1926, 24, 647—654, 655—667, 668—676).—Hydrastine may be determined in fluid extracts of *Hydrastis canadensis*, after removal of ethyl alcohol, by precipitation at the b.p. and acid reaction with silicotungstic acid, the precipitate being washed with 0·5% hydrochloric acid, dried, and ignited. The determination may also be made by conductometric titration (cf. B., 1925, 1011) with silicotungstic acid. In the titration method two curves are obtained, one by using 2 c.c. of extract and 50 c.c. of water as diluent, and the other by using 2 c.c. of extract and 50 c.c. of 70% ethyl alcohol as diluent, 0·8—1 c.c. of 10% sodium acetate being added in each case. The difference between the abscissæ corresponding with the points of inflexion gives the amount of silicotungstic acid required for precipitation of the hydrastine present in the extract taken. Good agreement was obtained between the gravimetric and the volumetric methods in the case of two extracts. G. W. ROBINSON.

Aluminium as constructional material in the organic chemical industry. BUSCHLINGER. —See X.

Butyl alcohol and acetone from maize. KILLEFFER. —See XVIII.

Determination of caffeine. GOBERT.—See XIX.

PATENTS.

Preparation of methyl alcohol. SOC. CHIM. DES USINES DU RHÔNE (F.P. 581,175, 7.8.23).—A mixture of hydrogen, or a gas containing it, and methyl formate vapour is passed at normal pressure and at 100—180° over the catalyst containing copper outlined in E.P. 219,649 (B., 1925, 869). Thus a mixture of 132 g. of the ester and 25 litres of hydrogen per hour was passed through a tube containing broken pot treated with the catalyst, and at 160° a mixture of hydrogen, 60 g. of unchanged ester, and 72 g. of methyl alcohol resulted, the two former being once more employed. The yield was some 90% of that calculated from the formic acid. Water-gas may be used instead of hydrogen. Decomposition of the ester is more rapid, and can be performed in a smaller apparatus when this method is used than by earlier processes. A. COUSEN.

Preparation of formaldehyde. G. TRÜMPLER (Swiss P. 111,120, 26.6.24).—The aqueous vapour of methylamine together with oxygen and indifferent gas is led at below 400° over catalysts (large excess of oxygen being avoided), and the chief reaction occurring is $2CH_3\cdot NH_2 + O_2 = 2CH_2O + 2NH_3$. As catalysts the oxides of copper, manganese, iron, nickel, and cobalt precipitated on porous or finely-divided material may be used, or simply material with large surface action as pumice, diatomite, kieselguhr, or asbestos. In the first case the most suitable temperature is below 300°; in the second, above 300°. The formaldehyde is easily removed from the condensate of the cooled reacting gas, giving a yield of 60% of the methylamine employed, whilst the ammonia can be recovered. A mixture of 5—10% of methylamine vapour, 10% of steam, and 80—85% of air, passed at 300—320° over granular diatomite with a velocity of 0·4 litre/hr. per 1 c.c. volume of reaction chamber, decomposed 30% of the methylamine, giving 24% of formaldehyde, 3% of carbon dioxide, and 3% of other oxidation products. A. COUSEN.

Production of alkylresorcinols. SHARP & DOHME, Assees. of A. R. L. DOHME (E.P. 250,892, 6.7.25. Conv., 16.4.25).—1 pt. of an acylresorcinol is reduced with 2½ pts. of zinc amalgam, using 5 pts. of hydrochloric acid (*d* 1·16) and 6 pts. of water, with vigorous agitation, at 105°, for 10—12 hrs., the product being washed, and distilled *in vacuo*. The zinc amalgam is prepared from granulated zinc and 2% mercuric chloride solution. The acylresorcinols may be prepared in any way, but more especially from resorcinol, fatty acid, and zinc chloride according to E.P. 250,893 (cf. B., 1927, 59), when no special purification subsequent to distillation of the crude product is generally required.
B. FULLMAN.

Reduction of aromatic nitro-compounds. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 263,376, 21.4.26).—Aromatic nitro-compounds are suspended in solutions of aluminium salts (especially the chloride) and reduced to amines by the addition of iron, acid being added if required. The presence of aluminium results in separation of the iron, after separation of the amine as usual, in the form of finely-divided yellow ferric hydroxide, which, after washing and drying, is available as a pigment, either immediately or, in red shade, on calcination. The red calcined product becomes brighter and more yellow as the amount of aluminium present increases. The aluminium salt may be produced during the reduction by addition of metallic aluminium to the iron. Examples are given of the application of the method to nitrobenzene, α-nitronaphthalene, and *o*-nitrotoluene. B. FULLMAN.

Production of camphene from pinene hydrochloride. H. GAMMAY (E.P. 263,311, 7.1.26).—Pinene hydrochloride is converted into camphene free from chlorine and phenol by heating and stirring it under pressure with excess of hydroxides or carbonates of the alkali or alkaline-earth metals in the presence of less than 20% of the amount of phenol required by the equation $C_{10}H_{17}Cl + C_6H_5\cdot ONa = C_{10}H_{16} + C_6H_5\cdot OH + NaCl$.

Naphthols, organic acids and their salts, and resin acids may also be used. *E.g.*, 2500 kg. of pinene hydrochloride are heated with 2000 kg. of sodium hydroxide solution (*d.* 1·53) and 200 kg. of phenol under 6—8 atm. pressure, while stirring. B. FULLMAN.

Reduction of alkyl esters [methyl formate]. M. E. BOUVIER and E. BLANC, Assrs. to SOC. CHIM. DES USINES DU RHÔNE (U.S.P. 1,605,093, 2.11.26. Appl., 18.9.23. Conv., 7.8.23).—See F.P. 581,175; preceding.

Extraction of tartaric [acid] salts [from wine-lees] (E.P. 263,340).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Gelatin. X. [Sensitising action of thiosinamine.] H. H. SCHMIDT (Phot. Ind., 1926, 1016—1018).—Results are given of tests on three plates submitted by Sheppard :—(*a*) with no added thiosinamine; (*b*) and (*c*) with increasing amounts of thiosinamine added to the same emulsion as (*a*). The relative speeds increased from (*a*) to (*c*): the spectral sensitivity extended in all cases to 520 $\mu\mu$; gradation of (*a*) was flat and that of (*b*) and (*c*) was normal; clarity increased from (*c*) to (*a*), (*c*) being fogged. The grain size and distribution were different in the three cases. In this respect the results differ from those of Sheppard, and indicate that the thiosinamine does not only take part in surface reactions, but also affects the grain structure. Also Schmidt's theory of the splitting-off of halogen ions from the lattice, combined with deformation of the bromine electron orbits, cannot hold by itself. Plates were bathed in ammoniacal solutions of thiosinamine in an increasing series of concentrations, to form silver sulphide nuclei at the grain surfaces. With increasing thiosinamine concentration the speed progressively decreased, and the speed of the untreated plate was never exceeded. It is concluded that silver sulphide nucleus formation is not the cause of increased speed in Sheppard's plates. All the thiocarbamide–silver bromide complex would decompose in the first ripening process, and it is not clear why the emulsion gains its high speed in the subsequent digestion process, after washing, when no ammonia is present and no further gelatin is added. The sulphide nucleus theory cannot explain the marked increase in speed obtainable with acid emulsions. In general, it is concluded that thiosinamine does not give increased speed if it merely affects the grain surfaces, but only if it takes part in the actual formation of the grains. W. CLARK.

Comparison of *X*-ray and white light exposures in photographic sensitometry. R. B. WILSEY and H. A. PRITCHARD (J. Opt. Soc. Amer., 1926, **12**, 661—689).—The effects of white light and of *X*-rays on photographic films have been compared. The characteristic exposure–density curves were similar for both types of radiation, and the maximum density that could be developed was the same. Reversal due to over-exposure was produced in the films by both *X*-rays and white light, and was found to consist of a progressive decrease in the rate of development, but not a decrease of the amount of silver developable. S. BARRATT.

Sensitisation for the entire visible spectrum. B. H. CARROLL (J. Opt. Soc. Amer., 1926, **13**, 35—37).—A bath conferring particularly uniform colour sensitivity and unusually high stability and white light sensitivity is made up as follows for a 5 × 7 plate tank: distilled water, 1500 c.c; pyridine, 15 c.c.; 0·1% pinacyanol solution, 6 c.c.; 0·1% pinaflavol solution, 6 c.c. The pyridine and dye solutions are mixed together and then added to the water. Plates should be pre-washed for 5 min. in distilled water, then bathed for 1 hr. at not more than 15°. Not more than twelve plates should be bathed in the above quantity. Successful tests were made on Eastman 36 plates. L. F. GILBERT.

***X*-Ray spectrographic examination of the tanning of membranes and tendons.** KATZ and GERNGROSS.—See XV.

Spectrography of flames in a combustion engine. HENNE and CLARK.—See II.

PATENTS.

Photography. TECHNICOLOR MOTION PICTURE CORP., and E. A. WEAVER (E.P. 263,331, 8.2.26).—A method is described for producing stained relief images in which the image has a more gradual gradation in the high lights than in the half-tone portions for the same range of light intensities in the subject, so that the detail of the scene may be accurately reproduced in the more highly-reflecting and/or illuminated parts of the scene. The characteristic curve of the dyed image has a concave lower portion, and the image is in the form of a relief having a stratum of uniform thickness in addition to the image stratum. Further, the image has more gradual thickness gradations in the thinner portions than in the thicker portions. The result is produced by exposing the sensitised material to a uniform light in addition to the image-printing light, the uniform exposure being of an intensity calculated to give an image the gradations of which are represented by a characteristic curve in which the lower portion is located above a tangent to the central part of the curve. The film is under-exposed to the image light, and the uniform exposure is made with light of a colour readily absorbed by the emulsion. The uniform exposure may be effected through the back of the film, and should not substantially exceed the threshold exposure. W. CLARK.

Producing chromophotographs on paper and chromodiapositives. F. JANZEN, Assr. to W. VOBACH (U.S.P. 1,613,515, 4.1.27. Appl., 14.3.23. Conv., 16.3.22).—See E.P. 195,056; B., 1924, 618.

XXII.—EXPLOSIVES; MATCHES.

Hydration of colloidal [propulsive] powders during drying. VIEILLE (Mém. Poudres, 1926, **22**, 132—142).—Determinations have been made of the loss of water, alcohol, and ether from colloidal powders during various methods of drying, and also of the amount of volatile matter still remaining in the powder. Stability tests at 110° were then carried out on the powders, and showed that "hydration" of the colloid was responsible for the loss in stability which followed treatment of the powder with hot water. After drying by drainage, about 90% of the residual volatile matter in the powder is water. A powder which was hydrated to excess by

steeping in alcohol and then allowed to drain in a saturated atmosphere could not be dried at 60° without a considerable loss in stability, and a steeping in hot water can have a bad effect on the stability.

S. Binning.

Loss of volatile matters from colloidal [propulsive] powders. Vieille (Mém. Poudres, 1926, **22**, 107—124, 125—131).—Tests on numerous types of French ordnance powders show that the loss of volatile matters takes place in three stages. First comes a rapid rate of loss, due to loss of moisture, followed by further loss at a slower rate caused by escape of solvent vapours. These two phases are separated by an intermediate stage, the nature of which varies with the type of powder. The phenomena are substantially the same whether the tests are carried out at 110°, 75°, 60°, or 40°, but the rate of loss naturally decreases with decreasing temperature, the loss in hours at 110° being practically equal to the loss in days at 75°, and probably equal to the loss in months at 40°. When a powder is heated at 110°, the percentage loss, after 4 hrs., represents the moisture, and the percentage losses from 4 to 20 hrs. or from 4 to 44 hrs. furnish a coefficient for the ease with which the particular powder loses it volatile matter. When heated at 110°, a French ordnance powder of satisfactory stability can give a coefficient of 0·6—0·9. There is no relation between the coefficients of loss of volatile matters and the stabilities at 110°, unless the coefficients fall below about 0·5 for strip powders, and to 0·6—0·7 for thicker powders. The chief cause of change in the ballistic properties of powders during storage is loss of moisture. A coefficient of 0·75 is suitable for powder during manufacture.

S. Binning.

Nitration of cellulose. Krauz and Blechta.—See V.

Patents.

Granulating ammonium nitrate. E. M. Symmes, Assr. to Hercules Powder Co. (U.S.P. 1,613,334, 4.1.27. Appl., 8.11.24).—Ammonium nitrate is converted into granules having an internal void space by spraying it at 115—160° into a gaseous cooling medium. S. Binning.

Ammonium nitrate explosive. E. M. Symmes, Assr. to Hercules Powder Co. (U.S.P. 1,613,335, 4.1.27. Appl., 8.11.24. Cf. preceding abstract).—The explosive contains a liquid explosive, carbonaceous material, and ammonium nitrate in the form of substantially spherical grains with internal cavities.

S. Binning.

Blasting methods and means. D. Farrell and A. W. Helmholtz (E.P. 262,941, 15.12.25).

XXIII.—SANITATION; WATER PURIFICATION.

Chloro-tastes [of water] and their eradication at Dallas, Texas. O. M. Bakke (J. Amer. Water Works Assoc., 1926, **16**, 730—736).—In one case, where the raw water was derived from a lake, chlorination of the raw water as well as the final effluent removed the tastes. Very satisfactory results were obtained when the residual chlorine of the raw water was maintained at 0·35 p.p.m., and that of the final plant effluent at 0·08—0·10 p.p.m. Tastes due to algæ could not, however, be eradicated in this manner. It was found necessary to control the algal growth by copper sulphate. In another case, taste was found to be due to the passage through old mains of chlorinated water deficient in bicarbonate. By reducing the lime dosage and producing a filter effluent having 30—40 p.p.m. of bicarbonate alkalinity and 10—20 p.p.m. of normal carbonate alkalinity, the objectionable taste was eliminated. W. T. Lockett.

Direct titrimetric methods for magnesium, calcium, and sulphate ions, and their application in water analyses. E. P. Schoch (Ind. Eng. Chem., 1927, **19**, 112—115).—By using thymolphthalein as indicator, and operating at 90—100° with a neutral solution containing magnesium, calcium, and sulphate ions, and such non-interfering ions as sodium, potassium, and chloride, the magnesium ion may be determined by titration with a standard lime solution, calcium with standard sodium carbonate solution, and sulphate by adding a standard barium chloride solution in excess of the sulphate present and titrating this excess with standard sodium carbonate solution. Thymolphthalein at 90—100° begins to turn blue with an alkalinity corresponding to p_H 10·23, and the titration of magnesium is completed with an alkalinity slightly less than that with which the colour appears. Precipitation of calcium carbonate is only complete when a definite blue colour is obtained, about half as deep as the standard. It is essential to remove hydroxyl ions from foreign sources (particularly those resulting from the titration of magnesium between neutral point and p_H 10·23) by neutralising. Boiling reduces the dissolved carbon dioxide to a practically definite minimum. The titration of the excess of barium chloride is complete only when the colour has reappeared and passed the standard. Details are given for the preparation of samples, and for typical analyses, including determination of sulphate ion alone, of permanent hardness or of sodium carbonates in natural waters, and of amounts of lime and soda required to soften water.

D. G. Hewer.

Patents.

Removal of air from water. E. Piron (U.S.P. 1,612,218, 28.12.26. Appl., 10.7.22. Renewed 4.3.23).—The water is brought into contact with a fixed air-absorbing gas at a temperature materially less than 100° by passing the water and gas in opposite directions through a distilling column. D. Woodroffe.

Chlorination of water. B. T. Brooks, Assr. to Mathieson Alkali Works Corp. (U.S.P. 1,613,438, 4.1.27. Appl., 22.3.24).—Liquid chlorine is vaporised by passing it through a heat exchanger in indirect relation with the liquid, the flow of gas being regulated by the amount of liquid chlorinated, after which it is introduced into the liquid. H. Royal-Dawson.

Exterminating injurious forms of life and composition therefor. H. Maxwell-Lefroy, Assr. to Graesser Monsanto Chemical Works, Ltd. (U.S.P. 1,613,402, 4.1.27. Appl., 15.6.25. Conv., 13.5.25).—See E.P. 261,055 and 261,241 ; B., 1927, 94.

// BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 11, 1927.

I.—GENERAL; PLANT; MACHINERY.

Adhesives and adhesion: true [organic] chemical compounds as adhesives. J. W. McBain and W. B. Lee (Proc. Roy. Soc., 1927, A, 113, 606—620; cf. B., 1925, 292; 1926, 291).—Pure crystalline organic substances may produce joints between optically-polished metal surfaces the breaking strength of which approaches 1 ton per sq. in., and are thus comparable as regards tensile strength with well-known adhesives. A number of substances were investigated, the metal test-pieces and the substance being heated to a temperature just above the m.p. of the latter, and the test-pieces being then "wrung" together. The strength of the joint is in some cases greatly increased by heating the substance (*e.g.*, saligenin) many degrees above its m.p. This may be due to various causes, such as resinification, or increase of viscosity due to loss of water of crystallisation. Coumarin is a good example of a definitely crystalline compound yielding strong joints. Tests with a number of organic liquids showed that they yield results of a lower order of magnitude, even when the film of liquid is only a few millionths of an inch thick. The highest results were obtained by wringing the surfaces together (cf. Budgett, Proc. Roy. Soc., 1912, A, 86, 25). Most of the substances yielding strong joints contained hydroxyl or carbonyl groups. In all cases, the thinner the film the stronger the joint, provided that the film is complete. Under similar conditions, joint strength with a given adhesive increases with the elasticity, the elastic limit, the tensile strength, and the hardness of the metals employed, and decreases with increasing atomic volume and compressibility. There is frequently a close parallelism between joint strengths in tension and in shear, and it is found that good lubricants with high spreading coefficients are poor adhesives. In explanation of the fact that disorderly molecular arrangement greatly enhances strength and joint strength, it is suggested that crystal grains in pure substances are joined to each other by true adhesion, which in this instance differs slightly but distinctly from the true cohesion of the same substance in the undercooled liquid or amorphous state.

L. L. Bircumshaw.

Patents.

Grinding mills. J. Jameson and R. Gaunt (E.P. 262,161, 2.7.25).—The material is fed to the space between stationary and rotary grinding discs and issues from between the peripheries of the discs. The spindle of the stationary disc is splined in a fixed support, and is adjusted axially through a worm and worm wheel, the latter engaging as a nut a thread on the spindle. Means are provided for locking the stationary disc in adjusted position.

H. Holmes.

Pulverising machine. C. C. Trowbridge (U.S.P. 1,609,529, 7.12.26. Appl., 5.11.25).—A rotary shaft extending into a drum carries spider arms provided with rollers which are freely rotatable against the drum wall. The interior of the spider communicates through an opening in one end of the drum with a casing enclosing an exhaust fan, and means are provided for driving the fan and the spider-carrying shaft independently.

H. Holmes.

Gyratory crusher. H. S. Anderson (U.S.P. 1,609,594, 7.12.26. Appl., 1.8.25).—A conical crushing head is carried by one end of a shaft which is gyrated by means engaging its other end. The shaft is provided, adjacent to the head, with a universal joint supported by a sleeve adjustable in the main frame.

H. Holmes.

Grading of powdered material by elutriation or by hydraulic classification. L. Andrews (U.S.P. 1,610,725, 14.12.26. Appl., 17.3.25. Conv., 25.3.24).—An ejector jet of water collects the material from the outlet of a bin, and the mixture passes on to a vortex chamber which has two outlets. The coarse material from one outlet passes to a settler, and the overflowing water regulates a float which causes an intermittent opening and shutting of the water-supply valve.

B. M. Venables.

Refrigeration. B. C. von Platen and C. G. Munters, Assrs. to Electrolux Serviel Corp. (U.S.P. 1,609,334, 7.12.26. Appl., 18.8.26).—A liquid cooling agent (liquid ammonia) is evaporated with consequent cooling in presence of an auxiliary agent (hydrogen), and a head is produced due to the difference between the vapour density of the mixture and that of the auxiliary agent. This head serves to force the mixture into a liquid (water) for absorbing the cooling agent, and to return the auxiliary agent thus separated into the presence of the vaporising cooling agent. The solution is heated to expel the cooling agent from the absorption liquid, the former being condensed and returned to the evaporator, and the latter being returned into the presence of the mixture.

H. Holmes.

Removing free moisture from substances. G. H. Elmore and R. C. Comley (U.S.P. 1,609,933, 7.12.26. Appl., 24.8.23).—The partly congealed or crystallised substance to be dried is continuously fed into and discharged from a centrifugal dryer. The treated material on leaving the rotor travels through the air a distance sufficient to enable the free moisture to dissipate by congelation and evaporation, and is received at an acute angle upon an arresting or collecting surface.

H. Holmes.

Products obtained by desiccation. W. H. Dickerson, Assr. to Industrial Waste Products Corp. (U.S.P.

1,609,993, 7.12.26. Appl., 3.11.22).—To prepare a solid organic substance in a dry, finely-divided state, it is stirred with water to produce a freely-flowing liquid mixture which is broken up into small particles. These are then dried by directing a current of drying gas into intimate contact with them. H. Holmes.

Separation of the constituents of gaseous mixtures. C. C. Van Nuys, Assr. to Air Reduction Co., Inc. (U.S.P. 1,612,164, 28.12.26. Appl., 26.1.23).—A portion of the cold compressed mixture is partially expanded, performing external work, and is subjected to selective liquefaction. Another portion is liquefied by indirect contact with colder media. The liquids thus obtained are mixed, and the mixture is rectified by direct contact with the partially-expanded gases. H. Holmes.

Apparatus for creating a vacuum. S. Percival. From Ken-Crip Corp. (E.P. 263,445, 17.6.25).—Air is admitted by a small port to a hollow member mounted within and communicating with a main casing in which a pump maintains a partial vacuum. A second hollow member surrounds the first, and the space between them is provided with an inlet from the atmosphere and a valved outlet to the main casing. Air admitted through this space into the casing upon opening the valve exerts an aspirating action on the first hollow member to maintain a partial vacuum therein. H. Holmes.

Metallic filter for air cleaner. H. H. Garner (E.P. 263,391, 26.5.26).—The filtering medium is a mass of wire crimped or curled and matted together, and preferably flattened in the planes of the crimps or curls. The wire, saturated with oil, is placed in a wire cage filling the upper portion of a casing. The lower portion of the casing is provided with a detachable bottom constituting a receptacle for oil. The air is admitted tangentially to this lower portion, and is drawn off from the top of the upper portion. The air in the lower portion produces a vortex of oil which separates the heavier dust particles, and the lighter particles are retained by the filter. H. Holmes.

Determining the specific heat of fluids. H. N. Packard, Assr. to Cutler-Hammer Manuf. Co. (U.S.P. 1,609,423, 7.12.26. Appl., 28.5.18).—A stream of the fluid and a stream of another fluid of known sp. heat are subjected to precisely similar heating influences. The relative quantities of the fluids required to enable each stream to undergo a like change of temperature are determined, and the required sp. heat is calculated therefrom. H. Holmes.

Disintegrating or pulverising apparatus. J. E. Kennedy (U.S.P. 1,609,298—9, 7.12.26. Appl., [A] 16.12.19, [B] 16.10.22).—See E.P. 204,250; B., 1923, 1160.

Apparatus for the heat treatment of materials [*e.g.*, tar]. A. M. Duckham, Assr. to Thermal Industrial & Chemical (T.I.C.) Research Co., Ltd. (U.S.P. 1,610,078, 7.12.26. Appl., 17.1.24. Conv., 2.2.23).—See E.P. 215,095; B., 1924, 543.

Rotary kiln for burning cement, ore, etc. (E.P. 262,525).—See IX.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Macroscopic constituents of Campine coals. M. de Boosere (Fuel, 1926, 5, 522—527).—Samples of three Campine coals (sub-metabituminous according to Seyler's classification) were separated into their macroscopic constituents, vitrain, durain, and fusain. Clarain was not obtained, either because of inability to distinguish it from vitrain or because it occurred in too finely divided zones. Each constituent was examined in the following particulars:—Density, proximate and ultimate analyses, solubility in chloroform and pyridine, the nature of distillation products up to 500°, the nature of the semi-coke produced at 500°, the degree of swelling, and the agglutinating index. The results are in accordance with those found for British coals. It is important in the sampling of coal to crush the coal completely before "quartering," as the constituents differ very materially in their hardness. S. Pexton.

Examination of Indian coals. B. Rassow and R. C. Bhattacheryya (Brennstoff-Chem., 1926, 7, 250—253, 264—268).—Indian coals, though geologically comparatively young, include lean coals, giving a powdery coke and only small yields of tar and gas, caking coals and gas coals, giving a hard coke, together with relatively high yields of tar. Previous data regarding Indian coals are meagre, but 22 samples are here described, containing 4—27% of ash, 15—37% of volatile matter, 0·56—1·5% of sulphur, and 1—1·8% of nitrogen. Four samples were carbonised at 500—550°, the tar yield ranging from 5—16%, and the tar itself containing 50—65% of neutral compounds, 35—50% of acidic compounds, and 1·5—2% of bases. The neutral oils boiling at 20—60° are composed entirely of paraffins, and in the higher fractions tetrahydrobenzene and its homologues are present, together with solid paraffins, but no aromatics. Phenol is absent from the acidic components of the tar, which include cresols, xylenols, and their homologues. The bases include pyridine and quinaldine. The aqueous condensate from the low-temperature carbonisation has an acid reaction, and contains pyrocatechol and ammonia, whilst the gas is comparatively rich in methane and poor in hydrogen. W. T. K. Braunholtz.

Reactivity of coke as a function of the oil-bitumen content of the original coal. R. Mezger and F. Pistor (Gas- u. Wasserfach, 1926, 69, 1061—1066).—The percentage loss of weight, referred to the ash-free material, of 0·1 g. of a carefully prepared sample of coke (200/250 mesh), when heated at 700° in a current of air for 5 min., is taken as a measure of its reactivity. Cokes are classified into groups of different reactivity according to the limits within which the loss of weight falls. Whether prepared in the laboratory or in a full-scale plant cokes from the same coals fall into the same reactivity groups. There is a close correspondence between the reactivity of a coke and the oil-bitumen content of the original coal (Fischer, Broche, and Strauch, B., 1925, 233), the former increasing as the latter decreases. This relationship is ascribed to the deposition of graphite of low reactivity, produced by the decomposition of the oil-bitumen. Coals of high bitumen content show a marked fall in reactivity with rising temperature

of carbonisation. Cokes prepared at 700° or over show no change in reactivity on being maintained for longer periods at the coking temperature. At 900° the oil-bitumen is completely decomposed and the graphite deposition reaches a maximum; cokes prepared at that temperature therefore do not change in reactivity even if further heated at 1000°. A. B. Manning.

Determination of the calorific value of fuels. II. W. Steuer (Brennstoff-Chem., 1926, **7**, 375—381; cf. B., 1927, 33).—It is unnecessary to line the bombs with platinum or other acid-resisting metal, an enamel lining being equally durable, provided precautions are taken (*infra*) to avoid spurting of red-hot particles of fuel against the lining. The leads to the ignition wire may be of stout nickel, and fine nickel wire is almost as good as platinum and preferable to iron wire for ignition purposes, as it melts without oxidation. The capsule in which combustion takes place is preferably made of a bad heat conductor, *e.g.*, quartz. It is unnecessary to remove all air from the bombs before admitting oxygen, since atmospheric nitrogen will not combine with oxygen under the conditions obtaining. The combustion of the fuel sample in briquette form gives unreliable results, owing partly to a tendency to sudden disintegration and consequent spurting, and partly to incomplete combustion, this being particularly marked with fuels of high ash content. It is preferable to leave the fuel in powder form and to wrap it round with waxed paper of known calorific value. Corrections for sulphuric and nitric acids produced in the bombs are unreliable, and should be omitted. The net calorific value may be determined directly, without knowing the hydrogen and moisture content of the fuel, by placing in the bomb a small nickel vessel containing phosphorus pentoxide or aluminium hydroxide, which is weighed before and after the combustion, the moisture being completely absorbed, under the conditions of the experiments, in 1 hr. by phosphorus pentoxide and 3 hrs. by aluminium hydroxide. W. T. K. Braunholtz.

Removal of tar from producer and coke-oven gas by electrostatic precipitation. J. Weyl (Stahl u. Eisen, 1926, **46**, 1863—1870).—The construction and principles underlying the operation of a Lurgi electrostatic precipitation plant are described briefly, and the results of a number of tests on the removal of tar and oil from producer and coke-oven gas by the apparatus are recorded. The gas passes directly from the producers to two precipitation chambers in series, then through three cooling towers, and finally through two more precipitation chambers. The temperature in the first two chambers is maintained at 80—90° to prevent precipitation of moisture in the tar deposited; almost complete removal of the tar is effected here, and the product contains less than 1% of water. In the cooling towers most of the water vapour is condensed, and a layer of mobile oil is obtained free from tarry matter, whilst the second series of chambers removes the last traces of mist and recovers a small quantity of light oil. The purified gases contain usually much less than 0·02 g./m.^{3} of suspended matter. The power consumption of the plant is less than one-quarter that of the usual centrifugal washing apparatus. A. R. Powell.

Distillation of oil-chalk in a current of heated gas. K. Hassel (Brennstoff-Chem., 1927, **8**, 5—8).—Experiments on the distillation of the bituminous chalk found near Heide in Holstein are described. By distilling the material at 420° in a vertical retort heated internally by the passage of a current of hot inert gas the oil is completely removed with a minimum of decomposition. For material containing 18% of oil and 3% of water the gas, an oxygen-free flue gas, enters at a temperature of 520° and leaves, with the products of distillation, at about 150°. The material has a tendency to cake, but no difficulty is found in passing the gas current through it. The retort, or at least the upper part of it, should, however, be lined with wrought iron to prevent sticking. The oil is of better quality, and in particular possesses a higher lubricating oil content than that obtained by distillation in an externally-heated rotary retort. A. B. Manning.

Synthesis of petroleum hydrocarbons. F. Fischer (Brennstoff-Chem., 1927, **8**, 1—5).—A *résumé* of the methods of transforming coal into oil is followed by a discussion of recent progress in the synthesis of paraffin hydrocarbons by the hydrogenation of carbon monoxide under ordinary pressures (cf. Fischer and Tropsch; B., 1926, 475). The difficulties of purification of the water-gas and regeneration of the catalyst have been overcome. The temperature of the cobalt or iron catalyst must be kept lower than that favourable to the production of methane, the necessary reaction rate being attained by suitable activation of the catalyst. The efficient production of benzine offers more difficulty than that of "gasöl" (a mixture of ethane, propane, and butane) or of the solid paraffins. The latter are produced when a strong base is added to the catalyst. The yield amounts to 100 g. of solid, liquid, and easily liquefiable hydrocarbons per cub. m. of water-gas. A. B. Manning.

Utilisation of light petroleum and petroleum residues. O. Aschan (Chem.-Ztg., 1927, **51**, 4).—The light petroleum fraction (b.p. 10—70°), consisting mainly of pentane and hexane, is suitable for use as a selective solvent and precipitant for many compounds, and as a source of derivatives of the lower paraffins. Thus the hydrocarbons can be used, by intermediate conversion into their monochloro-derivatives and suitable fractionation, for the production of, *e.g.*, amyl alcohols, or of technical amyl acetate. The residues of b.p. above 300° obtained from petroleum oils with a high naphthene content, *e.g.*, Russian masut, yield on pyrogenic decomposition at 700—800° considerable quantities of isoprene, benzene, and toluene. L. A. Coles.

Patents.

Apparatus for washing coals. P. Wolf (E.P. 254,709, 25.6.26. Conv., 3.7.25. Addn. to E.P. 206,151; B., 1925, 162).—The washing water pulsates by means of a piston in, or by the supply of water under pressure to, an independent chamber which communicates with the washing chamber proper only through the grids upon which the material to be washed settles. The washing chamber is under atmospheric pressure, and the cleaned coal passes over a weir the height of which controls the thickness of the layer of coal undergoing treatment.

According to the amount of schists in this layer the resistance to pulsation and the pressure of water in the base of the washing chamber vary. This variation is shown by the height of a float operating in a tube having its upper end open to the atmosphere and its lower end extending into the washer below the grids. The float operates a self-closing valve which connects the schist-discharging chamber with the atmosphere. When the valve is closed this chamber is air-locked, and no water carrying schist enters from the weir opening at the base of the bed of coal under treatment. When the valve is opened schist is washed into the discharging chamber from the coal layer until it is freed from excessive amounts; the valve then automatically closes.

S. PEXTON.

Fuels. J. M. W. KITCHEN (U.S.P. 1,606,239, 9.11.26. Appl., 9.6.24).—A coked fuel is impregnated with a liquid hydrocarbon, and any portion volatile at ordinary temperatures is removed. A. C. MONKHOUSE.

Manufacture of fuel bricks. R. TORMIN (U.S.P. 1,611,616, 21.12.26. Appl., 20.5.25. Conv., 28.5.24).—Coal-like fuel containing bitumen is mixed with a solvent for the bitumen and then compressed.

F. G. CROSSE.

Peat fuel production. L. BROWN and W. L. BROWN (U.S.P. 1,599,952, 14.9.26. Appl., 20.9.24).—Peat is fed into a stream of products of combustion from a combustion chamber, and the dry granular peat is conveyed by a fan to a container. A. C. MONKHOUSE.

Coking process. F. W. SPERR, JUN., Assr. to KOPPERS CO. (U.S.P. 1,605,378, 2.11.26. Appl., 12.9.18. Renewed 15.10.24).—The bituminous material to be coked is first liquefied and then fed into the top of a vertical retort containing the material which is being coked. The temperature is increased as the material descends, and the coke is withdrawn at the bottom.

A. C. MONKHOUSE.

Carbonising or gasifying fuel. INTERNATIONAL COMBUSTION ENGINEERING CORP., Assees. of W. RUNGE (E.P. 242,623, 27.10.25. Conv., 6.11.24).—Finely powdered fuel is carbonised or gasified by allowing it to gravitate slowly down a vertical retort up which hot oxidising gases pass. The gaseous heating medium is generated by the complete or partial gasification of the carbonised product in an expanded section of the retort at its base. The hot fuel gas produced is withdrawn from the top of the retort. S. PEXTON.

Manufacture of highly active carbon. E. URBAIN, Assr. to URBAIN CORP. (U.S.P. 1,610,399, 14.12.26. Appl., 29.3.26).—Cellulosic material, impregnated with a substance of acid reaction comprising oxygen and phosphorus in chemical combination, is heated at 700° until there is a substantial evolution of phosphides.

H. ROYAL-DAWSON.

Coke ovens. KOPPERS CO., Assees. of J. VAN ACKEREN (E.P. 252,310, 29.8.25. Conv., 23.5.25. Addn. to E.P. 230,167; B., 1925, 345).—The one "bus" flue for collecting the products of combustion from the tops of the vertical flues of a coke oven is replaced by a number of shorter "bus" flues situated at the level of the oven crown. Each flue is confined to five combustion chambers, and is provided with a central outlet port communicating with a common collecting flue at a higher level. Individually the shortened "bus" flues carry only a proportion of the combustion products from each heating wall, and therefore their cross-section can be reduced. This reduces the heating surface on the oven crown, and thus avoids excessive cracking of the volatile products, at the same time allowing the use of combustion flues of the maximum height. Maximum control of heating is possible with the minimum of dampers in this system.

S. PEXTON.

Process and apparatus for distilling oil shale. D. J. L. DAVIS and G. W. WALLACE, Assrs. to S.E. Co. (U.S.P. 1,607,240—1, 16.11.26. Appl., 20.9.22. Cf. U.S.P. 1,491,290; B, 1924, 503).—A vertical retort is fitted with a tapered bottom to which is fixed a pipe curving to the horizontal. The shale is fed upwards through the pipe. A zone of combustion is produced at the top of the retort, and maintained by passing in air through the retort cover. The oil distils off from the shale below this zone, trickles downwards, and is drawn off. The spent shale is continuously removed from the top of the retort. W. N. HOYTE.

Method and apparatus for the cracking of hydrocarbon oils. B. B. SCHNEIDER, Assr. to STANDARD OIL Co. (U.S.P. 1,609,000—1, 30.11.26. Appl., 12.6.25).—The oil is passed through a heating coil, where it is brought to cracking temperature; it passes to an enlarged chamber in which this temperature is maintained. The vapours evolved from this chamber pass up a dephlegmator, at the top of which they are cooled by passage over pipes carrying a current of oil used as cracking stock. Unvaporised oil is withdrawn from the large chamber. W. N. HOYTE.

Cracking of [petroleum] oils. G. L. PRICHARD and H. HENDERSON, Assrs. to GULF REFINING Co. (U.S.P. 1,607,966, 23.11.26. Appl., 6.5.22).—Oils of high b.p. are mixed with aluminium chloride and heated by hot oil in a heat interchanger. Cracking takes place.

W. N. HOYTE.

Cracking [hydrocarbon] oils. F. A. HOWARD, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,612,289, 28.12.26. Appl., 14.2.21).—The cracking stock is distilled under pressure, the vapours are mixed at atmospheric temperature with natural gas gasoline, the heavier portions of the vapours being first condensed; after a second condensation the vapours are scrubbed with a suitable heavy oil, the resulting oil together with the oil from the first condensation pass into contact with the vapours evolved from the still, and thence into the still.

W. N. HOYTE.

Petroleum still. F. I. DU PONT, Assr. to DELAWARE CHEMICAL ENGINEERING Co. (U.S.P. 1,609,349, 7.12.26. Appl., 9.11.23).—The petroleum to be distilled is floated on top of molten metal, the temperature of which is maintained by circulating a portion in an external pipe system in which heat is applied to the metal.

W. N. HOYTE.

Petroleum still. M. ROWE (U.S.P. 1,610,594, 14.12.26. Appl., 17.9.24).—The still comprises three concentric chambers; hot flue gases pass through the central chamber, the petroleum to be distilled passes

through the outermost chamber, and the vapours evolved therefrom pass via a dome through the chamber separating flue gases from petroleum. W. N. Hoyte.

Breaking oil–water emulsions. F. W. Harris, Assr. to Petroleum Rectifying Co. of California (U.S.P. 1,609,546, 7.12.26. Appl., 19.11.25).—Solid particles are added to the emulsion; by the use of an electric current the water agglomerates round the particles, thereby breaking the emulsions. W. N. Hoyte.

Breaking petroleum emulsions. M. De Groote, Assr. to W. S. Barnickel & Co. (U.S.P. 1,612,180, 28.12.26. Appl., 29.4.26).—Water-in-oil emulsions are treated with a chemical demulsifying agent; when they show a tendency to break they are subjected to electrical dehydration. W. N. Hoyte.

Dehydration of oil. J. Primrose, Assr. to Power Specialty Co. (U.S.P. 1,611,370, 21.12.26. Appl., 28.6.23).—A portion of the wet oil is heated in a vessel under pressure; pressure is then reduced, and the vapours evolved are condensed in a coil immersed in a further portion of the wet oil awaiting treatment. W. N. Hoyte.

Recovery of hydrocarbon oils from oil sands. H. H. Armstrong (U.S.P. 1,607,977, 23.11.26. Appl., 15.5.22).—The sand is treated with natural gas whereby separation of the oil in the sand can be obtained by gravity. W. N. Hoyte.

Hydrogenation of [petroleum] oils. A. McD. McAfee, Assr. to Gulf Refining Co. (U.S.P. 1,608,328—9, 23.11.26. Appl., [a], 13.1.22; [b], 27.3.22).—(a) An asphaltic petroleum is mixed with aluminium chloride and heated in the presence of a gas rich in hydrogen until the asphalt disappears. (b) An unsaturated gasoline by heating under pressure with the same gas and catalyst becomes hydrogenated. W. N. Hoyte.

Method and apparatus for desulphurising and fractionally separating petroleum. M. Benton, Assr. to Canadian American Finance & Trading Co., Ltd. (U.S.P. 1,607,043, 16.11.26. Appl., 7.4.24).—The petroleum is mixed with superheated steam, and the mixture is immediately allowed to expand. Hydrocarbons are fractionally precipitated with the water, and the fractionation is increased by spraying the vapours with hydrocarbon condensate. W. N. Hoyte.

[Fractional] distillation of oils. D. R. Weller and L. Link, Assrs. to Standard Development Co. (U.S.P. 1,609,007, 30.11.26. Appl., 12.11.20).—Vapours from a still are fractionally condensed, the condensates run separately through coils of pipes immersed in the still, and the vapours passing uncondensed through the succession of condensations are separately condensed and collected. W. N. Hoyte.

Refining of hydrocarbon oils. G. Egloff and H. P. Benner, Assrs. to Universal Oil Products Co. (U.S.P. 1,608,089, 23.11.26. Appl., 20.8.20. Renewed 9.6.26).—The oil is distilled in the presence of fuller's earth and caustic soda. W. N. Hoyte.

Production of gasoline and other hydrocarbons. B. Ormont, Assr. to Bernard Ormont Associates, Inc. (U.S.P. 1,608,664, 30.11.26. Appl., 27.5.21).—The lighter portions of the oil are vaporised by direct application of heat, and the heavier portions by the generation of nascent steam within the oil. W. N. Hoyte.

Apparatus for the abstraction of gasoline. C. H. Brown (U.S.P. 1,607,942, 23.11.26. Appl., 5.12.23).—The high-gravity gasoline is first abstracted from the gas by treatment with absorber oil, the gas is then cooled and subjected to a second treatment with the absorber oil already partially saturated with gasoline. The gasoline is then separated from the absorber oil by distillation. W. N. Hoyte.

Apparatus for cooling hydrocarbon vapours or gases at a high temperature. A. A. F. M. Seigle (U.S.P. 1,611,082, 14.12.26. Appl., 11.8.21. Conv., 25.7.18).—See E.P. 185,140; B., 1922, 849 a.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Acetate silk. R. O. Herzog (Papier-Fabr., 1927, **25**, 17—18).—Cotton linters are preferred as raw material not on account of their superior molecular aggregate, which is artificially lowered in the subsequent operations, but because of their freedom from β-celluloses etc., which tend to render the product harsh and horny. Wood pulps are used in minor quantities, but only after treatment for the removal of inferior cellulose constituents. The acetylation is essentially a heterogeneous reaction because the distribution and diffusion of the acetylating agent is not rapid and uniform throughout the process, and the primary acetate is not homogeneous; this drawback is largely corrected in the secondary treatment. The size of the molecular aggregate is considerably reduced, and higher dispersion ensured during this secondary reaction, and the acetyl value is also reduced, *i.e.*, free hydroxyls are developed; owing to these differences the solubility is modified and greater chemical homogeneity is obtained. The secondary acetate is far more responsive to plastifying agents than the primary, but no ideal plastifier, such as camphor with nitrocellulose, is known. The solution in acetone etc. is ready for spinning as soon as it is prepared; there is no secondary change in aggregation of the nature of "ripening." Both dry and wet spinning processes are practised with recovery of solvents. The dry process has the advantage of yielding directly and rapidly a strong, well-constructed thread (good Röntgen diagram of regenerated cellulose). In wet spinning quick coagulation gives poor threads; the best results are yielded by the "stretch" spinning process wherein the filaments are gradually coagulated and drawn out from relatively large orifices. The thread is water-repellent, and special adsorption methods of dyeing have been developed. Acetate silk possesses very special characters of lustre and handle.

Cellulose esters of aromatic sulphonic acids. G. Kita, T. Nakashima, and I. Sakurada (Cellulose Ind. Tokyo, 1926, **2**, 405—413).—The acid content of the cellulose ester formed by the action of *p*-toluenesulphonyl chloride on alkali-cellulose is uninfluenced by the temperature of the reaction between 8° and 30°, but at 30—80° the product contains a lower proportion of acid. Addition of excess of acid chloride has no effect on the

composition of the ester, but reduces the time necessary for reaction. The esterification proceeds further in the presence of an excess of alkali, but, with given concentrations of alkali and toluenesulphonyl chloride, a definite maximum esterification is reached. In this behaviour, and in the fact that there is no sudden liberation of heat, the reaction differs from those between alkali-cellulose and carboxylic acid chlorides. Alkali-cellulose prepared by the action of a 15—35% (by vol.) alkali solution yields with toluenesulphonyl chloride an ester containing 1 mol. of acid to 2 mols. of $C_6H_{10}O_5$. If an excess of alkali is added before esterification, the product consists principally of the monoester, and if the esterification is repeated 5 times the acid content of the product corresponds with that of a mixture of the mono- and the di-esters. An ester with a higher acid content is difficult to obtain even by esterification in presence of pyridine, under which conditions carboxylic acid chlorides give di- and tri-esters. An ester with a higher acid content than is required for the monoester is more stable towards heat than one of lower acid content. Similar results to the above were also obtained with benzenesulphonyl chloride. W. J. Powell.

Ripening of alkali-cellulose. D. Krüger (Cellulosechem., 1927, **8**, 1—3).—In the preparation of viscose, ripening takes place in the mercerised cellulose during the time which elapses before carbon disulphide is introduced. The length of time, temperature, and other conditions of the immersion are known to influence the properties of the resulting viscose, and diffusion experiments have shown that it is accompanied by a decrease in the size of the cellulose aggregates (B., 1926, 45). A method of following this change consists in carefully nitrating the alkali-cellulose under standard conditions, and determining the relative viscosity of the acetone solution of the nitrocellulose produced. The conditions of nitration must be standardised, as the nitration process itself causes a change in the size of the cellulose particles. A mixed acid containing 61·5% of sulphuric acid, 20% of nitric acid, and 18·5% of water masked the differences due to varying times of immersion in alkali, whereas an acid containing 71·7% of sulphuric acid, 18·7% of nitric acid, and 9·6% of water gave more satisfactory results. Samples were withdrawn at intervals from cellulose undergoing the alkaline treatment with the following results: duration of immersion (hrs.), 1·5, 28, 76, 118; copper number (Braidy method), 1·0, 0·8, 0·7, 0·8; relative viscosity of acetone solution of the corresponding nitrocellulose (1·5 g. in 100 c.c.), 20·8, 12·7, 8·4, 6·4. A cellulose from another source gave: duration of immersion (hrs.), 1·5, 4·5, 26·5, 52·5, 94·5; copper number, 1·0, 1·0, 0·9, 0·8, 0·8; relative viscosity, 18·0, 14·1, 12·0, 10·4, 8·2. The method therefore gives an indication of the degree of ripening of the sample under investigation. W. J. Powell.

Reducing power of sulphite liquor and of lignosulphonic acid. F. Kurtz (Cellulosechem., 1927, **8**, 3—5).—The total organic matter in sulphite liquor is greater than the amounts of lignosulphonic acid and fermentable sugars present, the difference being due to the presence of polysaccharides which should, on submitting the liquor to hydrolysis, yield further quantities of fermentable sugars. The increase in reducing power, as indicated by the copper number (Bertrand method), on boiling sulphite liquor or lignosulphonic acid with dilute mineral acids is, however, small. Boiling with alkalis causes a reduction in copper number. The best results are obtained when the liquor is digested for 2 hrs. with 2% sulphuric acid at 120—130°, a treatment which increases the copper number of sulphite liquor from 29·4 to 36·6, and that of lignosulphonic acid from 2 to 5·6. W. J. Powell.

Chemical properties and relative value of spring and autumn wood in manufacture of sulphite pulp. E. Hägglund and T. Johnson (Papier-Fabr., 1927, **25**, 22—23, and Zellstoff u. Papier 1927, **7**, 49—50). —The analyses of the separated spring and autumn layers of wood showed only insignificant differences in the chemical constituents, the principal divergence being in the pentosans: 7·58% for the spring wood and 6·77% for the autumn. Digestion experiments carried out in glass tubes gave equivalent yields of cellulose, except when a liquor rich in lime was used for a slow digestion. In that case the spring wood gave 2% more cellulose than the autumn. In all cases liquors from the spring wood were poorer in sugars than those from the autumn wood. The autumn wood produced more volatile organic acids. The chemical constants of the pulps from both kinds of wood were equivalent for equivalent digestions. The mechanical tests showed a higher tensile strength and elongation for the autumn wood cellulose, but the folding tests were distinctly better for the spring-wood cellulose; hence the autumn wood yields a stronger but more brittle fibre. J. F. Briggs.

Lignin. K. Kürschner-Brünn (Cellulosechem., 1927, **8**, 5—7).—Chiefly polemical. The author considers that lignin, as isolated from wood by any of the biological or chemical processes now in use, is not identical with the natural substance, as is shown by the difference in its behaviour towards solvents. Boiling trichloroacetic acid dissolves considerable quantities of lignin, and the chloroacetylated products, precipitated from the solution by addition of water, yield vanillin when heated with alkalis. W. J. Powell.

Erratum.

Pectin contents of flax. The work attributed to W. Kind in the abstract on p. 911 of the B Abstracts of Nov. 11, 1926, was actually done by W. Honneyman (J. Text. Inst., 1925, **16**, T 370; cf. B., 1926, 187). The notes by Kind in Spinner u. Weber (not Spär- u. Wirtsch., as given in the abstract) were merely extracts from Honneyman's paper.

Patents.

Production of high-grade half-stuff. H. Wenzl, Assr. to I. G. Farbenind. A.-G. (U.S.P. 1,611,969, 28.12.26. Appl., 8.7.26. Conv., 25.1.26).—Spinning-mill waste, flax, hemp straw, or jute is digested at a raised temperature with an aqueous solution of 1 pt. of a monosulphite to 3—3·5 pts. of a bisulphite, with the addition of a soluble neutral salt. B. P. Ridge.

Producing a material similar to vulcanised fibre. K. Zarfel (E.P. 261,959, 15.2.26).—Lignified vegetable fibres (cellulose) are boiled for 2—3 hrs. in a closed vessel with dilute caustic soda solution to open

them. The boiled mass is washed free from lye, rinsed in clean water, ground, and moulded. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 20,613 of 1899, 7058 of 1889, and 8493 of 1886.)

D. Woodroffe.

Manufacture of lustrous fabrics. F. Wolf, Assee. of H. Kühn (E.P. 256,244, 28.7.26. Conv., 1.8.25).—Lustrous fabrics closely resembling glacé thread are made by treating the fabric with a finishing composition and passing it while moist and in a stretched condition in close contact with a number of cylindrical brushes rotating rapidly in the direction of the warp threads and in the direction of travel of the fabric. Heating cylinders may, if desired, operate between the brushes. A suitable composition contains 80 litres of water, 10 kg. of wheat starch, 0·5 kg. of paraffin, and 0·5 litre each of Turkey-red oil and glycerin. D. J. Norman.

Waterproofing textile and other materials. C. J. Moreton, and Waterproofers (Moreton's Process), Ltd. (E.P. 262,605, 24.12.25).—The material is soaked in an emulsion consisting of palm oil, tallow, ammonia, glycerin, borax, wax, and Karaya gum, afterwards treated with a 2% aqueous solution of titanium sulphate, zinc sulphate, or alum, and subsequently dried.

B. P. Ridge.

Manufacture of artificial silk. Spinnstoff-Fabr. Zehlendorf G.m.b.H. (E.P. 260,872, 29.4.26. Conv., 21.12.25).—Minute cavities of uniform size are produced in viscose threads by subjecting the spinning solution to electrolysis. Pointed electrodes are used to ensure the formation of small gas bubbles, and are located immediately before the spinning nozzles so that the thread is spun before the small bubbles can unite into larger ones of irregular size. The amount of gas developed can be accurately controlled by adjusting the intensity of the current. Little or no oxygen appears at the anode owing to the presence of readily-oxidisable sulphur compounds in the viscose solution; this has the advantage of reducing the quantity of hydrogen sulphide formed during the spinning operation. D. J. Norman.

Manufacture of artificial silk. M. Hirasawa, Assr. to S. Hoshino (U.S.P. 1,603,080, 12.10.26. Appl., 27.4.25. Conv., 7.5.24).—Fibrous material consisting chiefly of fibroid is dissolved in zinc chloride solution and spun into filaments; these are passed successively through a solution of an alkali hydrogen sulphite and a solution of formaldehyde in, *e.g.*, alcohol.

D. J. Norman.

Manufacture of artificial silk. K. Leuchs (U.S.P. 1,611,354, 21.12.26. Appl., 12.4.26. Conv., 16.12.25).—Artificial silk bands or plates are produced by electrolysing the viscose solution supplied to a nozzle, electrodes being placed across the flow, and the viscose being afterwards formed into threads. B. P. Ridge.

Production of thin foils and film-like bands from soluble cellulose derivatives. A. Eichengrün (E.P. 241,590, 20.10.25. Conv., 20.10.24).—Concentrated solutions of cellulose derivatives are spread on one side, or both, of a quickly travelling, pliable, endless band which moves horizontally until solidification commences and is then led vertically in the form of long loops over rotating discs or wheels which come into contact with the edges of the band only. Drying is finished in this loop-way, the films are removed, and the band is returned for a fresh application of solution. Films 0·03—0·01 mm. thick may thus be produced at a rate of 5—10 m./min. B. P. Ridge.

Making artificial sponges. L. Mostny (U.S.P. 1,611,056, 14.12.26. Appl., 21.11.25. Conv., 25.2.25).—A solution of cellulose ester is mixed with pore-forming material, the mixture treated with steam from an external source, and the pore-forming material finally separated. B. P. Ridge.

Production of cellulose sheets. J. Feith and J. W. Ziegler, Assrs. to J. C. Patten (U.S.P. 1,611,175, 21.12.26. Appl., 8.12.21).—Cellulosic sheets are obtained by dissolving cellulose material in a cuprammonium solution, filtering, expelling ammonia from the filtrate, spreading it on a smooth surface, and allowing it to set to a compact film, which is then dried, washed with water and dilute acid, removed from the surface, and dried under tension. Water-white transparent sheets are produced if a solution containing at least 5% of cellulose is used and the film given a further acid and water wash after removal from the surface and before drying under tension. B. P. Ridge.

Treatment of the black liquor obtained in the manufacture of wood fibre by the soda process. E. Hägglund (E.P. 258,035, 4.7.25).—Increased yields of by-products such as acetone, methyl alcohol, and acetates are obtained from soda-cellulose black liquor by adding alkali, *e.g.*, 4% of caustic soda on the weight of wood treated, prior to the removal of most of the organic constituents in a carbonaceous form by heating the waste liquor at 350° under a pressure of about 150 atm. After separation of the carbonaceous matter, the liquor is preferably divided into two portions, about 75% being returned, after causticising, to the digester, whilst the remainder is concentrated (with recovery of methyl alcohol) and dry-distilled to recover acetone. Alternatively, the whole of the white liquor may be causticised, during which the methyl alcohol may be collected and a portion treated for the recovery of sodium acetate by concentration and crystallisation, the mother-liquor being subsequently returned to the digester. The quantity of liquor diverted for this purpose should be such that the sodium acetate content of the digestion liquor as a whole remains fairly constant.

D. J. Norman.

Method and apparatus for forming products from pulpy mass. H. Buel (E.P. 260,362, 5.8.25).—Pulp containers are made by forcing a suspension of pulp at a consistency of 0·3—1% into a perforated mould under a pressure of 40—125 lb./sq. in. gauge pressure. The pulp layer is compacted by a gaseous medium under pressure with simultaneous reduction of pressure on the outside of the mould, pulsations of pressure being induced to improve the felting of the fibres. Drying is effected by pumping an inert gas, such as nitrogen or carbon dioxide, at a temperature of 135—540° under a pressure of 75—125 lb./sq. in. or more, into the mould while maintaining a vacuum outside the mould. Waterproofing agents may be

distributed over the inner surface of the dry container and forced into the mass by heat and pressure.
D. J. NORMAN.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

PATENTS.

Production of cyanic compounds. O. STÄLHANE (U.S.P. 1,610,897, 14.12.26. Appl., 31.3.26. Conv., 18.11.24).—A charge of briquettes consisting of carbon and an alkali compound is fed continuously through pipes heated in an electric furnace, and in which passes a counter-current stream of nitrogen.
H. ROYAL-DAWSON.

Removal of borax from alkali nitrates. C. F. BOOTH and P. LOGUE, Assrs. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,610,485, 14.12.26. Appl., 28.7.24).—Lime and magnesia are added to the solution of dissolved nitrate to precipitate the borax. H. ROYAL-DAWSON.

Method of chlorinating solutions. J. H. MACMAHON, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,609,755, 7.12.26. Appl., 23.1.24).—Chlorine, chiefly in the liquid state, is introduced directly into the stream of solution to be chlorinated through a circulating pump, thus causing an intimate admixture within the pump. H. ROYAL-DAWSON.

Apparatus for the evaporation of liquid chlorine. J. H. MACMAHON, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,609,756, 7.12.26. Appl., 8.11.24).—Liquid chlorine is passed under pressure through an expansion valve into an evaporating vessel supplied with a water-jacket kept at atmospheric temperature, means being provided for heating the water when the temperature drops below that of the atmosphere.
H. ROYAL-DAWSON.

Apparatus for chlorinating solutions. J. H. MACMAHON, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,609,757, 7.12.26. Appl., 8.12.24).—The apparatus consists of a tank for the solution, connected by a pipe line to which is attached a circulating pump for withdrawing the solution and returning it to the tank. Liquid chlorine under pressure passes through a pipe fitted with reducing valve directly into the circulating solution. In this manner it is intimately mixed with the solution. H. ROYAL-DAWSON.

Process and apparatus for preparing bleach liquors. J. H. MACMAHON, Assr. to MATHIESON ALKALI WORKS (U.S.P. 1,609,758, 7.12.26. Appl., 24.12.24).—Liquid chlorine is released under reduced pressure, and the gas is introduced into a portion of an alkaline solution which, after being well agitated, is pumped together with unabsorbed gas through a closed passage back to the remainder of the solution.
H. ROYAL-DAWSON.

Clarification of bleach liquors. J. H. MACMAHON, Assr. to MATHIESON ALKALI WORKS (U.S.P. 1,609,759, 7.12.26. Appl., 10.3.25).—Suspended coarse mineral matter including lime is separated from the bleach liquor, which is then chlorinated until the remaining finely divided lime dissolves. The chlorination is not carried beyond neutrality. H. ROYAL-DAWSON.

System for collecting radium emanation. G. FAILLA (U.S.P. 1,609,614, 7.12.26. Appl., 24.1.23).—A vessel containing radium compound is provided with a space for the collection of radium emanation in a partial vacuum over a relatively long time, means for purifying the emanation, and a variable mercury level which automatically withdraws successive portions of the emanation and transfers them to the purifier in a relatively short time. J. S. G. THOMAS.

Deoxidiser. C. J. RODMAN and J. G. FORD, Assrs. to WESTINGHOUSE ELECTRIC & MANUF. CORP. (U.S.P. 1,610,592, 14.12.26. Appl., 21.12.23).—Pulverised material capable of spontaneous combustion consists of several oxidisable constituents, of which one is powdered carbon and another is copper.
H. ROYAL-DAWSON.

IX.—BUILDING MATERIALS.

PATENTS.

Waterproof composition [cement]. R. ILLEMANN (E.P. 262,961, 31.12.25).—Stone flour (or powdered mineral matter) is mixed to a creamy fluid with water, the mixture is boiled, and liquid bitumen or mineral wax is added, with agitation, until a pasty mass is obtained, forming an elastic waterproof cement on cooling.
B. W. CLARKE.

Production of hydrated lime. W. E. CARSON (U.S.P. 1,613,341, 4.1.27. Appl., 4.4.24).—Quicklime is ground to a fineness greater than 100 mesh and mixed with sufficient water to react with all the lime to form a dry hydrate without a rise in temperature above 110—115°. F. G. CROSSE.

Wood preservation. L. C. DREFAHL, Assr. to GRASSELLI CHEMICAL Co. (U.S.P. 1,612,102, 28.12.26. Appl., 15.4.25).—Wood is preserved by impregnation with a solution of a toxic organic arsenic compound in paraffin. A. DAVIDSON.

Rotary kilns for burning cement, ore, or similar materials. VICKERS, LTD., and L. D. PARKER (E.P. 262,525, 12.9.25).—The shell of a rotating kiln is extended beyond the usual lower end, and ports are formed through the cylindrical wall of the extension through which the burnt material passes to a number of smaller cylinders surrounding and rotating with the main shell. The further ends of the cylinders are left open and the outgoing material is cooled by incoming air. The open ends are partly closed by baffles which prevent the material falling out of any cylinder when it is above the furnace. B. M. VENABLES.

[Portable] mixing machines [for concrete]. D. WHITAKER (E.P. 262,518—9, 11.9.25).—A concrete or other batch mixer is provided (in A) with wheels of special form and (in B) with means for working the aggregate from the centre of the rotating bowl to the circumference, comprising a pyramidal central projection and fixed scraping blades. B. M. VENABLES.

Cement, concrete, and plaster [containing rubber]. V. LEFEBURE (U.S.P. 1,610,864, 14.12.26. Appl., 29.2.24. Conv., 5.3.23.).—See E.P. 217,279; B., 1924, 676.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Determination of carbon in cast iron. J. T. Mackenzie (Iron Age, 1926, **118**, 415—416; Chem. Abstr., 1926, **20**, 3663).—The oxygen is passed through 4-mesh soda-lime containing 15% of water, asbestos, and then dry 12-mesh soda-lime; in the combustion tube it is passed through an alundum thimble, over the sample on a bed of ferric oxide, then through oxidised copper gauze, and finally through another alundum thimble containing asbestos. It is then passed through a bulb containing loosely-packed glass wool and glass beads in separate chambers. Stopcocks permit the introduction and withdrawal of concentrated sulphuric acid saturated with chromic acid. The carbon dioxide is absorbed with "ascarite." A. A. Eldridge.

Sulphur in iron and steel. J. Ciochina (Chim. et Ind., 1926, **16**, 889—901).—Samples of iron taken at the time of tapping at various points between the furnace and the receiving vessel for the molten iron showed a decrease in sulphur content from 0·031% at the furnace to 0·011% at the receiving vessel at the end of 20 min. and also with duplicate samples the one allowed to cool with free access of air had only half the sulphur content of that cooled in an enclosed space. The sulphur in these cases is not eliminated as sulphur dioxide, for carbon, although more combustible, is not thus reduced in quantity. Neither is the decrease in sulphur due to a migration of sulphur in the molten material from below to the surface, for in samples taken at the beginning of tapping, at the middle, and at the last stage the sulphur content was found to be the same throughout the melt. That the desulphurisation is not due to a difference in the distribution of sulphur between the two solvents, metal and slag, was proved by disturbing the equilibrium by the addition of a large amount of caustic soda to the slag; samples before and after showed very little variation in sulphur. The author has studied the possibility of desulphurisation of iron and steel with ferro-manganese, and gives some 24 curves with various irons and steels showing that as the manganese is increased the sulphur decreases. The whole process in a Martin furnace has been studied from the time of fusion to that of tapping, and cases have been found where the sulphur content increased, due, not to the materials used, but to the sulphur in the producer gas. W. G. Carey.

Corrosion of ship plates and rivets. W. Bennett (Engineering, 1926, **122**, 796—799).—The causes underlying corrosion of rivets in ship plates are discussed, and emphasis is laid on the probable electrolytic nature of the phenomenon. The higher the copper content the greater may be the corrosion-resisting nature of the steel. Iron rivets are generally electro-negative to ship plates, and are less corrosive than steel rivets. L. M. Clark.

Granulation of slags. B. Bogitch (Compt. rend., 1926, **183**, 1291—1292).—Additional precautions to the author's method of prevention of explosions during the granulation of slags (B., 1926, 194) are :—(1) The molten slag should fall into the centre of the granulating tank to avoid cracking the walls. (2) Certain cobaltiferous slags may explode when they touch the water before the air-jet can come into effect owing to their high viscosity and conduction of heat. The temperature of the furnace should therefore be raised at the last moment to keep them fluid, and quenching should be carried out in warm water at 35—40°. J. Grant.

Elastic hysteresis of some alloys [brass, duralumin, and nickel steel]. L. Jannin (Rev. Mét., 1926, **23**, 709—717).—When a rod of an alloy, such as brass, duralumin, or 2% nickel steel, is subjected to a series of progressively increasing loads, all of which are applied for 2—3 min., and the extensions at the beginning and end of the time are plotted against the load, a smooth curve is obtained until the elastic limit is reached. At this point, and for every successive increment of load thereafter, a small, but gradually increasing, extension takes place during the time of application of the load. This phenomenon of elastic hysteresis indicates that the metal is in a semi-plastic, semi-elastic state. If the metal is allowed to recover after having been strained to this state, and then the experiment is repeated, the elongation continues to be elastic long after the true elastic limit has been passed. When, however, this metal, after two extensions, is submitted to compression carried out in a similar way, the deformation under constant charge reappears after a certain lower elastic limit has been passed, but again disappears if the metal is once again subjected to compression while in the semi-plastic state. The lower elastic limit characterises the resistance of the metal to alternating stress, and the higher limit the resistance to unidirectional stress. In the case of a mild 2% nickel steel the lower elastic limit is raised by annealing, quenching, and tempering at 250°. A. R. Powell.

Corrosion products and mechanical properties of certain light aluminium alloys as affected by atmospheric exposure. E. Wilson (Proc. Physical Soc., 1926, **39**, 15—25).—Measurements have been made on the electrical conductivities, the corrosion products, and tensile properties of aluminium (99·55%) and certain aluminium alloys during exposure to London atmosphere during 25 years. The alloys contained more than 98% Al, together with various amounts of iron, silicon, copper, nickel, manganese, and zinc. A gradual increase in resistance takes place with pure aluminium mainly due to loss of metal. With 0·16% Cu corrosion is reduced, but with larger amounts the alloy slowly disintegrates. Both the zinc and nickel alloys showed a remarkable variation in electrical conductivity during exposure. A slight fall in tensile strength took place with all specimens during the whole period. C. J. Smithells.

Cementation of aluminium by copper. J. Cournot and E. Pérot (Compt. rend., 1926, **183**, 1289—1291).—Cementation occurs if aluminium cylinders coated electrolytically with copper are heated to a temperature above 544°, the temperature of the solid solution eutectic Al_2Cu. Partial fusion is therefore necessary. The penetration increases with the temperature used, and the hardness increases also, until it reaches a maximum at the m.p. of aluminium, and then falls. The degree of penetration increases with the thickness of the copper deposit. Microscopical examination shows

that the penetration is eutectic in nature, small crystals of Al_2Cu being sometimes seen. It is inferred that copper diffuses into pure aluminium more slowly than the eutectic diffuses into the solid solution, and this is confirmed by the fact that penetration takes place to a greater extent in duralumin than in aluminium.

J. GRANT.

Silumin: mechanism of the "modification" process. J. CZOCHRALSKI (Z. Metallk., 1927, **19**, 14—16).—The various theories put forward to account for the modifying action of sodium on silicon–aluminium alloys are briefly discussed. The protective colloid theory of Otani (B., 1926, 831) is considered to be untenable as the crystals of aluminium are not decreased in size by the modification process; in fact, the more powerful the modifying action the coarser are these crystals. As a structure resembling that obtained by addition of sodium to silicon–aluminium alloys may be obtained by rapid quenching from just below the m.p., it is suggested that sodium simply affects the crystallisation constants of the molten alloy. A. R. POWELL.

Scleron and aeron. E. SCHEUER (Z. Metallk., 1927, **19**, 16—19).—Scleron is an aluminium alloy resembling duralumin, but containing lithium in place of all or part of the magnesium; it is characterised by a high resistance to change of shape, by a high tensile strength (40—50 kg./mm.^{2}) and hardness, and by a good elastic limit (20 kg./mm.^{2}) and limit of proportionality (30 kg./mm.^{2}). Aeron, on the other hand, is a copper–aluminium alloy which can be improved by ageing, and is characterised by great pliability, having an elongation of 18—25%, with a reduction of area of 30—40%, combined with a tensile strength of 36 kg./mm.^{2}, an elastic limit of 12 kg./mm.^{2}, and a limit of proportionality of 20—24 kg./mm.^{2}. Scleron thus resembles a mild steel and aeron wrought iron in mechanical properties. The sp. gr. of the alloys are 2·95 and 2·8 respectively, the coefficients of expansion $2 \cdot 6 \times 10^{-5}$ and $2 \cdot 36 \times 10^{-5}$, the thermal conductivities 0·24 and 0·36, and the electrical conductivities 16·7 and 24. The heat treatment of scleron comprises quenching from 480° and ageing at the ordinary temperature for a few days. The greatest hardness of aeron is obtained by ageing at 100—120° after quenching at 500°; both alloys are completely annealed again by heating at 300—350°. Aeron is a little more resistant to attack by sea water when aged below 100°, and much less when aged at higher temperatures than is commercial aluminium.

A. R. POWELL.

Lautal. FUSS (Z. Metallk., 1927, **19**, 19—21).—Lautal (4% Cu, 2% Si, remainder commercial Al) is readily forged at 400—500° and subsequently hardened by ageing at 120—130°. The sand-cast alloy has a tensile strength of 12—20 kg./mm.^{2}, an elongation of 4%, and a Brinell hardness of 55—60; after ageing the corresponding values are 16—26 kg./mm.^{2}, 4%, and 65—70. The chill-cast alloy has a tensile strength of 15—25 kg./mm.^{2}, an elongation of 10%, and a hardness of 66—80; after ageing these values become 20—30 kg./mm.^{2}, up to 10%, and 90—110 respectively. The alloy has been used satisfactorily for the framework of aeroplanes and motor boats and for the construction of the rotors of Flettner's rotor-ship. A. R. POWELL.

Constructal. W. SANDER (Z. Metallk., 1927, **19**, 21).—Constructal is an aluminium alloy containing only 3% of alloying elements, chiefly zinc, and, after ageing, has a tensile strength of 40 kg./mm.^{2}, an elongation of 22%, and a Brinell hardness of 118. By modifying the ageing conditions a tensile strength of 52 kg./mm.^{2}, with an elongation of 10—12%, or an elongation of 26—28% with a tensile strength of 36—38 kg./mm.^{2}, may be obtained. Another form of the alloy containing a somewhat larger proportion of other elements has a tensile strength of 60 kg./mm.^{2}, and a much better resistance to corrosion than ordinary aluminium, owing to the alloying elements being entirely in solid solution.

A. R. POWELL.

PATENTS.

Concentration of ores by flotation. H. W. ELLEY, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,610,217, 7.12.26. Appl., 24.4.25).—The ore in the form of a pulp is subjected to a flotation operation in the presence of an *s*-dialkylthiocarbamide.

M. E. NOTTAGE.

Froth-flotation concentration of ores. C. P. LEWIS, Assr. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,610,298, 14.12.26. Appl., 19.4.26).—An iron-bearing zinc ore may be concentrated by agitating a pulp of the ore with sufficient lime to render it alkaline and with a sulphur-containing organic compound, and further agitating it with a copper salt and a mineral-frothing agent. The froth, relatively rich in zinc and poor in iron, is then separated. M. E. NOTTAGE.

Method and apparatus for counteracting scaling and corrosion. H. J. CREIGHTON, Assr. to ANTI-SCALE CORP. (U.S.P. 1,611,853, 21.12.26. Appl., 4.10.24).—A metallic surface in contact with a fluid may be protected against the formation and adhesion of scale by including it in a metallic electric circuit through which a current flows, the voltage maintained in the circuit being below that which would cause electrolytic corrosion of the surface beyond what would normally occur in the absence of such voltage, and intermittently interrupting the current. M. E. NOTTAGE.

Manufacture of zirconium compounds. C. J. KINZIE, Assr. to THE TITANIUM ALLOY MANUF. Co. (U.S.P. 1,609,826, 7.12.26. Appl., 27.10.25).—Zirconium ores are mixed with an alkali and, without fusion, decomposed by heat to form a zirconium compound soluble in dilute acids. M. E. NOTTAGE.

Applying a plating of aluminium alloy to iron and steel sheets and bands. TRIERER WALZWERK A.-G. (E.P. 258,837, 9.7.26. Conv., 24.9.25. Addn. to 245,736).—Sheets or bands of an alloy of aluminium and magnesium with copper, manganese, or nickel may be combined with iron or steel sheets or bands by first rendering the surfaces metallically clean and bringing them together first between the rolls of a cold-rolling mill, uniting them solely by this pressure, and then converting them by repeated further cold rolling into a thin laminated strip. M. E. NOTTAGE.

Applying a metallic layer to another metal.

R. Walter (U.S.P. 1,609,747, 7.12.26. Appl., 31.10.25).—In the deposition of a metal layer by pouring it when molten upon another highly heated metal, the latter is preheated by continuous application of a reducing gas stream over that portion of it which is to be covered by molten metal. M. E. Nottage.

Process for coating metals. S. Daniels and A. C. Zimmerman (U.S.P. 1,608,775, 30.11.26. Appl., 23.7.25).—A coating of silicate solution applied to the metal is dehydrated and subsequently neutralised by treatment with a dilute acid solution. J. S. G. Thomas.

Manufacture of bi-metallic strips for thermostats. F. W. Miller (E.P. 262,644, 21.4.26).—Bi-metallic strips or plates for thermostats may be made by covering a plate of the metal of higher m.p., such as nickel steel, with a flux, *e.g.*, powdered fused borax, mixed with the metal of lower m.p., *e.g.*, brass, in a granular form. This again is covered with the metal of lower m.p., either as coarse grains or in the form of a plate, and heated to a temperature such that the upper metal is fused. On cooling, the metals adhere closely, and the bi-metallic plate may be rolled out to any desired thickness. Still better coherence may be obtained if the metal of higher m.p. be first cleaned and then etched with acid. M. E. Nottage.

Production of ferrophosphorus. E. V. Rawn (U.S.P. 1,613,125, 4.1.27. Appl., 12.4.26).—To obtain ferrophosphorus of high phosphorus content a ferrophosphorus low in phosphorus is treated in an electric furnace with material containing phosphorus and a flux. F. G. Crosse.

Manufacture of molybdenum steel. F. M. Becket, Assr. to Electro Metallurgical Co. (U.S.P. 1,613,571, 4.1.27. Appl., 19.11.25).—Molybdenum sulphide is introduced into a steel bath, the reaction taking place in the absence of abnormal reducing conditions. F. G. Crosse.

Alloy. H. S. Cooper, Assr. to Kemet Laboratories Co., Inc. (U.S.P. 1,610,262, 14.12.26. Appl., 11.2.25).—An alloy, chiefly composed of a metal having the properties of nickel and cobalt, also contains 1—12% W, 1—12% Ti, 4—10% Al, and 2—6% Si. M. E. Nottage.

Metallic composition. W. H. Judy, Assr. to Sumet Corp. (U.S.P. 1,611,043, 14.12.26. Appl., 24.6.24).—The composition consists of 1—20% Pb, 75—55% Cu, and 24—25% Zn. M. E. Nottage.

Alloy for welding copper. H. Canzler (E.P. 260,565, 5.10.26. Conv., 2.11.25).—The silver in an alloy of copper with silver may be replaced in part (up to 60%) by manganese. A satisfactory alloy consists of 0·6% Mn, 0·4% Ag, a trace (0·05%) P, and remainder copper. M. E. Nottage.

Stainless iron and durable pen alloys. M. Yonezu (U.S.P. 1,611,798—9, 21.12.26. Appl., 23.4.24).—The alloys contain 8—30% Cr, 1—10% V, and the remainder (A) iron, or (B) a metal of the iron group. F. G. Crosse.

Dental alloy. C. C. Vogt and J. W. Harsch, Assrs. to L. C. Smith & Son Manuf. Co. (U.S.P. 1,612,782, 28.12.26. Appl., 19.12.23).—An alloy containing silver and tin in amounts which will readily amalgamate with mercury, together with a small quantity of a non-ferrous metal of the nickel group. F. G. Crosse.

[Fusible] alloy. E. H. Richardson, Assr. to Edison Electric Appliance Co., Inc. (U.S.P. 1,612,151, 28.12.26. Appl., 11.8.22).—The alloy consists of a zinc base with less than 10% each of bismuth and aluminium. F. G. Crosse.

Effecting exothermic reactions. C. R. Schroeder, Assr. to Metal & Thermit Corp. (U.S.P. 1,609,970. 7.12.26. Appl., 31.10.23).—Ores and oxides of tungsten may be reduced by initiating the exothermic reaction with aluminium, and completing the reduction with a less active reducing agent. M. E. Nottage.

Rotary kilns for burning ores etc. (E.P. 262,525).—See IX.

XII.—FATS; OILS; WAXES.

China-wood oil. II. E. Fonrobert and F. Pallauf (Chem. Umschau, 1927, 34, 1—6).—A further contribution to the controversy regarding the mechanism of the webbed drying of tung oil (see B., 1926, 371). After a lengthy discussion of the various conflicting theories, further experiments are described to show the effect of light on the transformation of α-elæostearic glyceride into the β-modification. Test samples of tung oil when treated in the dark with catalytic amounts of sulphur and selenium in carbon disulphide, bromine and iodine in carbon tetrachloride, α- and β-naphthol, cresylic acid, and ethereal hydrochloric acid, show little change except in the case of that catalysed with selenium in carbon disulphide, when crystallisation of the β-form is complete in 30 days. In the light, transformation occurs in from 4 days with sulphur in carbon disulphide, to 27 days with iodine in carbon tetrachloride. Less action is found in the case of the naphthols and cresylic acid, whilst only with ethereal hydrochloric acid is no action apparent, and it is suggested that the β-elæostearic glyceride is sufficiently ether-soluble to prevent precipitation. Oxygen, sulphur, and selenium also accelerate this rearrangement. Only slight polymerisation with consequent thickening occurs during these tests. Experiments on the webbing of tung oil when heated are then described. Raw oil fails to dry in 24 hrs. at 15°; in 22 hrs. at 50° it dries hard, and with a good lustre, in ½ hr. at 110° the greater part dries highly wrinkled but partly smooth and glazed, whilst in ¼ hr. at 150° it gives a clear, highly glazed, but slightly gelatinous surface. A wood oil varnish containing 2·5% of a cobalt drier (having 4% Co) dries partly webbed and partly glazed at 15°, whilst at 50°, 100°, and 150° it is very webbed. These experiments were carried out in the dark and in an atmosphere of carbon dioxide. It is deduced that the webbing at the higher temperatures is brought about by quick surface oxygen-absorption giving a skin which increases in volume, and since the foundation is still liquid wrinkling sets in. At ordinary temperatures the webbing may be caused by a similar process, together with the formation of the β-elæostearic glycerin. A modification of the earlier theory of intramolecular polymerisation, re-esterification, and extramolecular polymerisation is put forward to explain the drying of tung oil, in which

each tung oil acid residue is assumed to have three, not two, double linkings. The new scheme of rearrangement is explained diagrammatically. The boiling of tung oil is discussed, and it is recommended to carry out the process by raising the temperature quickly, and for a minimum time, to 330—340°, and subsequently to cool very rapidly to prevent gelatinisation. (A number of corrections to Part I are included.) E. HOLMES.

Detection of castor oil in fatty mixtures. VIZERN and GUILLOT (Ann. Chim. analyt., 1927, [ii], 9, 1—2).—About 10 g. of the fatty mixture are saponified, and a mixture of the dry soap with 7—8 g. of caustic potash is heated in a porcelain crucible gently at first and then strongly, and the fused potash intimately mixed with the soap. On the appearance of white fumes heating is stopped, the crucible covered, and when cool the under surface of the lid will have the odour of octyl alcohol if 5% of castor oil was present in the original mixture. With practice and the use of a comparative test with the same predominating oil as in the mixture as little as 1% of castor oil can be detected. It is probable that the oil of *Curcas purgans* would give a positive reaction, but for typical non-drying, semi-drying, and drying oils negative results were always obtained. D. G. HEWER.

Use of colloidal earth in oil and fat splitting in the manufacture of soap. R. TEFS (Z. deuts. Oel-Fett-Ind., 1926, **46**, 801).—The addition of colloidal earth promotes emulsification and consequently saponification. In experiments in saponification with 2*N*-caustic soda without and with the addition of 1 g. of earth per 10 g. of oil, the degrees of saponification without and with earth, respectively, were for cotton-seed oil 59—100%, for linseed oil 60·2—100%, for soya bean oil 91—100%, and for palm oil 85·4—93·9%. With *N*-alkali the respective degrees of saponification were, for whale oil 68·3—75%, for Japanese fish oil 59·2—82·8%, for linseed oil 43—84·2%, for rape oil 40·8—60·2%, for olive oil 55·6—98·7%, for arachis oil 24·7—65%, and for tung oil 92·8—98·8%. It is possible to produce a clay soap directly from oil or from fatty acids which shall be more soluble, and lather more easily, than ordinary soap. A. RAYNER.

PATENTS.

Oil solvent process. J. EHRLICH (U.S.P. 1,610,270, 14.12.26. Appl., 9.2.25).—Non-polymerised oils are extracted from animal and vegetable oleaginous materials containing them, by nuclear halogenated aromatic hydrocarbons. S. S. WOOLF.

Process of treating liquids. L. CALDWELL, Assr. to CELITE Co. (U.S.P. 1,603,314, 19.10.26. Appl., 12.10.25).—Oleaginous liquids are dried by being brought into intimate contact with dry lime-treated diatomaceous earth, and then separating the treated earth. D. F. TWISS.

XVI.—AGRICULTURE.

Phosphoric acid content of crops grown on peat soils as an index of the fertilisation received or required. F. J. ALWAY, W. M. SHAW, and W. J. METHLEY (J. Agric. Res., 1926, **33**, 701—740).—Ash analyses of grain, straw, and hay from fertilised and unfertilised plots on peat soils in Minnesota are reported. The soils contained abundant supplies of lime, but were very deficient in phosphates. There was no consistent influence of fertilisation on the total ash content of the crops. The percentage of phosphoric acid in the dry matter was greatly increased by applications of phosphate, the increase being greatest where some unfavourable factor, other than supply of nutrients, caused a low yield. With straws, the increase in some cases amounted to several hundred per cent., with hays from 20—50%, and with grains from 10—60%. Definite limiting values for the P_2O_5 content of a crop were difficult to fix on these soils, since the figures varied greatly from year to year with different weather conditions. In cases where the use of potassium fertilisers increased the yields, the percentages of P_2O_5 in the dry matter were lowered; otherwise, potassium had no effect. C. T. GIMINGHAM.

Phosphoric acid needs of soils determined by colorimetric method; effect of calcium and iron. A. NĚMEC (Compt. rend., 1926, **183**, 1295—1297).—The presence of a considerable amount of calcium in soil renders inaccurate colorimetric determinations of phosphates on aqueous extracts of soil. Iron and aluminium in soil bind phosphoric acid in forms useless for the plant. L. F. HEWITT.

Microbiological analysis of soils as an index of soil fertility. X. Catalytic power of soil. S. A. WAKSMAN and R. J. DUBOS (Soil Sci., 1926, 22, 407—420).—The catalytic action of soils, as measured by the liberation of oxygen from hydrogen peroxide under standard conditions, has been studied. A neutral or slightly alkaline reaction is favourable and an acid reaction is unfavourable to this action. There is a certain parallelism between the crop-producing capacity of soils and their catalytic action. The determination does not, however, give definite information about the microbiological processes of the soil, since the liberation of oxygen is a result, not only of the action of catalase, but also of the catalytic action of organic substances present in abundance in soils rich in organic matter or of inorganic catalysts which are specially active in alkaline soils. C. T. GIMINGHAM.

Origin and nature of soil "humus." V. Rôle of micro-organisms in the formation of "humus" in the soil. S. A. WAKSMAN (Soil Sci., 1926, **22**, 421—436).—Experiments are discussed on the decomposition of straw, cellulose, and fungus mycelium in sand and soil, and the formation of "humus" therefrom. The results indicate that, when plant residues are added to soil, most of their constituents are decomposed rapidly, though some resist decomposition. Micro-organisms convert part of the carbon into microbial protoplasm, assimilating nitrogen at the same time. A part of this protoplasm may persist in the soil for some time, and contributes definitely to the soil "humus"; it is probably the important source of nitrogen in the "humus." Soil "humus" is made up of (1) lignins and other constituents of plants, and (2) certain ingredients of the protoplasm synthesised by micro-organisms. C. T. GIMINGHAM.

Relation of manganese and iron to a lime-induced

chlorosis. B. E. Gilbert, F. T. McLean, and L. J. Hardin (Soil Sci., 1926, **22**, 437—446).—Various crops suffer from chlorosis on certain limed soils at the Rhode Island Agricultural Experiment Station. The condition is not due to deficiency of iron or magnesium. Chlorosis is, in this case, associated with an abnormally low manganese content of the affected plants, and is remedied by applying very small amounts of manganese salts in the form of sprays. C. T. Gimingham.

Synthetic calcium silicates as a source of agricultural lime. III. Comparison of the influence of synthetic calcium silicates with other forms of lime on the soil reaction. R. M. Barnette (Soil Sci., 1926, **22**, 459—466).—It is concluded, from measurements of the p_H of soil suspensions, that chemically equivalent and practical applications of calcium carbonate, hydrated lime, dicalcium silicate, and "limosil" (a mixture of monocalcium silicate and calcium oxide) bring about approximately equal changes in the p_H of the soils to which they are added. C. T. Gimingham.

Electrometric and Hutchinson-MacLennan methods of measuring lime requirements of acid soils. C. Brioux and J. Pien (Compt. rend., 1926, **183**, 1297—1299; cf. B., 1914, 932).—The p_H of soil gives little indication of its lime requirements. In general, the Hutchinson and electrometric methods of determining the lime requirements of soil give the same results. The amount of lime required to alter the p_H of 1 kg. of soil by 0·1 at about p_H 8 is suggested as a "saturation coefficient" to indicate the buffering action of soils. Light, sandy soils have a low "saturation coefficient," whereas clay soils rich in humus give high results.
L. F. Hewitt.

Fertilising action of cyanamide and its derivatives. C. Brioux (Chim. et Ind., 1926, **16**, 883—888).—In a soil sufficiently moist the reduction of cyanamide to urea and of urea to ammonia is complete in 48 hrs., but with less moisture and, therefore, less contact with the soil polymerisation to dicyanodiamide takes place; the use of cyanamide as a fertiliser under cover is not advocated, but out of doors rain speedily dissolves the cyanamide and brings it into contact with soil organisms. Cyanamide cast in sticks and supplied in bags is specially liable to polymerisation, owing to its partial hydration and the pressure to which it is subjected in manufacture; samples of this were totally inactive as fertilisers and were even deleterious. The nitrogen present as dicyanodiamide should be tested for as well as the cyanamide nitrogen. Guanylurea is not harmful to the same degree as dicyanodiamide, but is equally inactive as a fertiliser for at least several months. A solution of free cyanamide treated with sulphuric acid is converted into urea sulphate, and this solution concentrated gives with natural phosphates a nitro-superphosphate which is preferable as a fertiliser to mixtures of superphosphate and cyanamide, for in the latter dicyanodiamide and guanylurea are formed. W. G. Carey.

Patents.

Preparation of fertilisers containing sulphur and phosphate. B. Bodrero (E.P. 262,017, 26.7.26).—Sulphur ore or by-products containing sulphur are heated so as to produce sulphur vapours which are condensed and sublimated in a mixer lined with lead or like substance, which permits the sublimation as well as the simultaneous incorporation of the sulphur with the finely powdered phosphate. D. Woodroffe.

XIX.—FOODS.

Patents.

Manufacturing artificial skins for sausages. J. Samuel (E.P. 262,202, 18.9.25).—Animal substances, *e.g.*, skin-splits, sinews, etc., are disintegrated, and worked with 15—40% of water to form a paste which is forced through a nozzle with an annular orifice into a heated drying room. F. R. Ennos.

Manufacturing artificial skins for sausages. J. Samuel (E.P. 262,352, 18.9.25. Addn. to 262,202, preceding).—Suitable mandrels, such as rotating cylindrical cores, are dipped into the paste previously described. The adhering film is dried at about 50° for a few hours and stripped off. F. R. Ennos.

Producing soluble cocoa powders. H. Bollmann (E.P. 262,239, 9.11.25).—Cocoa powder is mixed with 2% of phosphatides derived from vegetable materials, in particular soya beans. These substances, possessing an intense emulsifying effect, prevent the particles of cocoa and drops of melted fat from separating. If the phosphatides are purified by removing the main quantity of oil centrifugally and distilling under reduced pressure, the phytosterol is retained and the treated cocoa powder has higher food value. F. R. Ennos.

Preparation of a cattle food-stuff from herring or other fish, seal, and whalemeat, and the waste products thereof. A. Bu (E.P. 263,014, 26.4.26).—The finely-divided raw materials above mentioned, from which the water may be pressed out if desired, are mixed with molasses or treacle. F. R. Ennos.

Preparation of pineapple stock food. S. M. Corbett (E.P. 263,047, 29.6.26).—Pineapple residue, obtained as a pulp after the juice has been expressed from the shells, cores, trimmings, etc., is dried and mixed with wheat middlings, coconut or cotton seed meal, and dried animal tissue from which part of the fat has been extracted. F. R. Ennos.

Autoheterolysis of animal and vegetable substances. M. Kahn, E. le Breton, and G. Schaeffer, Assrs. to Soc. Franç. des Prod. Alimentaires Azotés (U.S.P. 1,611,531, 21.12.26. Appl., 16.6.25. Conv., 17.6.24).—See E.P. 235,834; B., 1925, 734.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

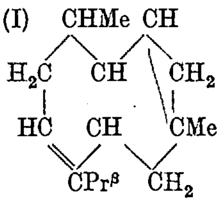
Toxicity of the arsenobenzenes. I. Methods of determining the arsenic in the organic molecule. G. Sensi (Annali Chim. Appl., 1926, **16**, 491—509).—The best method for determining arsenic in either salvarsan or neosalvarsan is that of Bressanin (A., 1911, ii, 1133), carried out as follows: 0·2—0·4 g. of the product is decomposed by heating with sulphuric acid for 2 hrs.; the cold liquid, freed from sulphur dioxide by means of a current of air, is made up to 100 c.c. with sulphuric acid (d 1·45). From this the arsenic is precipitated by addition of 2·5 c.c. of 30% potassium iodide solution, and the precipitate dissolved in water, super-

saturated with sodium hydrogen carbonate, and titrated with $0 \cdot 1N$-iodine solution. In the case of a compound salified by silver, the liquid freed from sulphur dioxide is diluted with 50 c.c. of sulphuric acid (*d* 1·45) and 2 c.c. of hydrochloric acid, and the silver chloride is removed by filtration through asbestos, which is washed with sufficient sulphuric acid to give 100 c.c. of liquid. The subsequent procedure is as given above. T. H. POPE.

Oil of turmeric. B. N. RUTOVSKII and P. P. LEONOV (Troud. Naoutchn. Chim.-Farm. Inst., 1924, [10], 36—48; Chem. Abstr., 1926, **20**, 3774).—The oil (1·03—1·42%, with loss of up to 9% of the santonin) had d_0^{25} 0·92111, $[\alpha]_D$ — 3·19°, n_D^{25} 1·4650, acid value 2·8, ester value 12·1, cineole 84·25%. Steam rectification, which causes a loss of 7·5% of cineole, gives an oil having d_4^{25} 0·9153, $[\alpha]_D$ — 2·64°, n_D 1·4627, acid value 1·8, ester value 12·3. The 0—85° fraction contains a little *d*-pinene. A. A. ELDRIDGE.

Constituents of oil of Supa. New natural source of copaene. G. G. HENDERSON, W. McNAB, and J. M. ROBERTSON (J.C.S., 1926, 3077—3080).—The chief constituent of Supa oil is the tricyclic sesquiterpene copaene, probably (I), which yields cadalene readily on heating with sulphur. The presence of a sesquiterpene alcohol is indicated in a fraction, b.p. 145—155°/10 mm., d_4^{18} 0·9484. Triacontane, $C_{30}H_{62}$, m.p. 62—63°, and a *compound*, m.p. 172—178°, are present in small quantity. B. W. ANDERSON.

(I)



Citrus oils. P. LIOTTA (Profum. ital., 1925, **3**, 340; Chem. Abstr., 1926, **20**, 3774).—Lemon oil had *d* 0·8643, $[\alpha]$ + 60·5°, citral 4·5%; bergamot oil, *d* 0·882, $[\alpha]$ + 14°, linalyl acetate 38%; mandarin oil, *d* 0·857, $[\alpha]$ + 71°, methyl anthranilate 0·6%; Portugal oil, *d* 0·850, $[\alpha]$ + 90·5°, aldehydes 1·3%; Seville orange neroli oil, *d* 0·8564, $[\alpha]$ + 91·3°, aldehydes 0·9%; Seville orange petitgrain oil, *d* 0·9009, $[\alpha]$ + 13°, esters 55·6%; lemon petitgrain oil, *d* 0·907, $[\alpha]$ + 18°, citral 18—19%; mandarin petitgrain oil, *d* 0·890, $[\alpha]$ + 11°, esters (as linalyl acetate) 53%; orange petitgrain oil, *d* 0·8854, $[\alpha]$ + 37°, aldehydes 6·5%; neroli oil, *d* 0·8852, $[\alpha]$ + 4·5°, esters (as linalyl acetate) 4%; cedrate oil (*Citrus cedra*), *d* 0·8692, $[\alpha]$ + 60°, aldehydes 4%; lime oil, *d* 0·8555, $[\alpha]$ + 58°, aldehydes 12%. A. A. ELDRIDGE.

Oil from the leaves and flowers of *Dictamnus fraxinella*, Pers. B. N. RUTOVSKII and I. V. VINOGRADOVA (Trans. Sci. Chem. Pharm. Inst. [Moscow], 1924, [10], 71—75; Chem. Abstr., 1926, **20**, 3774).—Oil from the flowers (0·05%) had d_4^{20} 0·9006, $[\alpha]_D$ + 20·97° in benzene. Oil from the leaves (0·15%) had *d* 0·9744, $[\alpha]_D$+ 1·04°, acid value 1·89, ester value 34·15, acetyl value 43·33, f.p. — 2°. Anethole and methylchavicole were present. A. A. ELDRIDGE.

Volatile oil of *Ledum groenlandicum*. E. V. LYNN, A. LEHMAN, and R. CAIN (J. Amer. Pharm. Assoc., 1926, **15**, 263—265; Chem. Abstr., 1926, **20**, 3778).—The oil (0·013%; 0·035% of parts other than stems) of Labrador-tea has $d^{21 \cdot 2}$ 0·8998, n_D 1·4917. Small amounts of phenols and aldehydes as well as sesquiterpenes and azulene are present, but very little ledum camphor or other stearoptene. A. A. ELDRIDGE.

PATENTS.

Manufacture of compounds of C : C-disubstituted barbituric acids and 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of R. MAYER (E.P. 245,107, 18.12.25. Conv., 24.12.24).—Colourless compounds of the above, having analgetic and soporific properties, are prepared by heating the components together in molecular proportions in air or in a carbon dioxide atmosphere, in the presence of an organic solvent, below 100°. The solvent is evaporated at room temperature *in vacuo*. The following *compounds* of the pyrazolone are described: with diethylbarbituric acid, m.p. 112—115°; with dipropylbarbituric acid, m.p. 93—95°; with phenylethylbarbituric acid, m.p. 127—130°; with *iso*propylpropenylbarbituric acid, m.p. 94—97°. B. FULLMAN.

Manufacture of new diacyl*iso*thiourea-*S*-alkyl esters. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 255,466, 14.7.26. Conv., 16.7.25).—Diacyl derivatives of *S*-alkyl-ψ-thiocarbamides are obtained when salts of the latter, in solution in tertiary organic bases (preferably pyridine), react with acyl chlorides. Examples are given of the preparation of the following derivatives of *S*-ethyl-ψ-thiocarbamide: the *dibenzoate*, m.p. 111—112°; the *di*-p-*nitrobenzoate*, m.p. 216°; the *diacetate*, an oil; and the *dicarbethoxy*-derivative, m.p. 45°. (Cf. A., 1903, i, 524.) B. FULLMAN.

Making salol and other esters. L. E. MILLS, Assr. to DOW CHEMICAL Co. (U.S.P. 1,606,171, 9.11.26. Appl., 19.1.25).—In making aryl esters by the action of phosphorus oxychloride on a mixture of a phenol and an acid, the salt of an alkali-forming metal and a readily volatilised acid is added. B. FULLMAN.

Solution of arsenobenzene derivatives. G. W. RAIZISS and A. KREMENS, Assrs. to ABBOTT LABORATORIES (U.S.P. 1,609,960, 7.12.26. Appl., 16.7.24).—A solution of an arsenobenzene derivative contains sodium thiosulphate, alone or mixed with a sugar, as stabilising and detoxicating ingredients. B. FULLMAN.

Preparation of substituted cyanamides. J. L. OSBORNE and G. BARSKY, Assrs. to AMERICAN CYANAMID Co. (U.S.P. 1,611,941, 28.12.26. Appl., 29.6.25).—Aromatic substituted cyanamides are prepared by treating cyanogen chloride with an aqueous solution of an aromatic amine. A. DAVIDSON.

Methods of producing amines, the substitution products thereof, nitriles, and tetrazoles. KNOLL & Co., and K. F. SCHMIDT (E.P. 250,897, 23.9.25).—Aromatic hydrocarbons or carbonyl compounds react with 1 mol. of hydrazoic acid in the presence of catalysts of an acid nature to form amines and their substitution products. Aldehydes under the same conditions yield nitriles. With an excess of hydrazoic acid under these conditions, carbonyl compounds yield the corresponding 1 : 5-disubstituted 1 : 2 : 3 : 4-tetrazoles. As catalysts, phosphorus pentoxide, chlorides, and oxychloride, zinc, ferric and aluminium chlorides, etc. may be used. *E.g.*, 20 pts. of anhydrous ferric chloride are slowly added to a cooled and stirred solution of

35 pts. of hydrazoic acid in 500 pts. of benzene, 15 pts. of acetone are then added drop by drop. The benzene is distilled off, the residue dissolved in water, iron precipitated by sodium hydroxide, and the altered solution shaken with benzene to separate the 1 : 5-*dimethyl*-1 : 2 : 3 : 4-*tetrazole*, m.p. 71°. *cyclo*Hexanone similarly yields 1 : 5-*pentamethylene*-1 : 2 : 3 : 4-*tetrazole*, m.p. 58—59°. Again, benzaldehyde treated with excess of hydrazoic acid in presence of stannic chloride yields a mixture of benzonitrile and *phenyltetrazole*, m.p. 69°. The use of concentrated mineral acids as catalysts is covered by E.P. 252,460 and 257,418 (B., 1926, 646, 932).

A. Davidson.

Production of lachrymating gases. D. B. Bradner, Assr. to Federal Laboratories, Inc. (Re-issue 16,495, 7.12.26, of U.S.P. 1,565,899, 3.5.24).—An effective concentration of lachrymatory gas in non-persistent form is produced from an organic compound not readily volatile at ordinary temperatures (such as a halogenated acetophenone), which lachrymates at concentrations below 0·01 mg. per litre in 3 min., mixed with a nitrated organic substance (such as nitrocellulose) which in burning vaporises the lachrymator without decomposing it. Decomposition of the mixture is prevented by addition of an anti-acid. B. Fullman.

Material for stimulating growth of hair. A. Weil, Assr. to H. C. Nagel (U.S.P. 1,608,686, 30.11.26. Appl., 12.10.25).—Stimulation is by the internal administration of hydrolysed keratin, split up into polypeptides and amino-acids, to which cystine has been added; or by the use of a completely hydrolysed keratin material in an ingestible container which is not substantially attacked by gastric juices, but which is readily dissolved by the intestinal digestive fluids. B. Fullman.

Centrifugal machines for the treatment of vaccine or similar substances. Nutriment, Ltd., and T. W. Tattersall (E.P. 263,046, 29.6.26).—The material, delivered to the inside of the rotor, passes outwards through radial or inclined grooves in a part of the rotor, and is thence discharged through grooves, preferably oppositely inclined, in a part of the stator, and two or more concentric pairs of such co-operating grooved parts of the rotor and stator are provided, the inner member of each pair receiving the material discharged from the outer member of the preceding pair. The engaging surfaces of the co-operating parts are preferably conical, and their relative positions are adjustable axially. The rotor shaft may be journalled in a tubular member fixed to the stator, the material being fed to the inside of the rotor through holes in the stator from an annular chamber surrounding the tubular member. H. Holmes.

Recovery of camphor and naphthalene contained in gaseous mixtures. J. H. Brégeat, Assr. to Brégeat Corp. of America (U.S.P. 1,613,218—9, 4.1.27. Appl., [a] 17.12.20; [b] 20.6.21. Conv., [b] 19.3.20).—See E.P. 150,654; B., 1920, 734.

Production of solutions of mercury derivatives of hydroxysulphobenzoic acids. E. Sagi, Assr. to Chemosan A.-G. (U.S.P. 1,613,569, 4.1.27. Appl., 13.8.23. Conv., 17.8.22).—See G.P. 399,394; B., 1924, 927.

Production of metaldehyde. T. Lichtenhahn, E. Lüscher, and H. Steiger, Assrs. to Elektrizitätswerk Lonza (U.S.P. 1,612,032, 28.12.26. Appl., 10.6.25. Conv., 23.6.24).—See E.P. 235,867; B., 1925, 738.

Making nitrogen-containing derivatives of terpene alcohols. R. Wolffenstein (U.S.P. 1,611,978, 28.12.26. Appl., 29.3.26. Conv., 2.7.23).—See E.P. 260,159; B., 1927, 28.

Compound of silver iodide and protein substances. T. Smith, Assr. to Parke, Davis & Co. (U.S.P. 1,610,391, 14.12.26. Appl., 27.9.20. Renewed 17.5.26).—See E.P. 206,954; B., 1924, 76.

Manufacture of emulsion of medicaments soluble in oil. M. Hartman and H. Kägi, Assrs. to Soc. of Chem. Ind. in Basle (U.S.P. 1,611,190, 21.12.26. Appl., 30.3.23. Conv., 8.5.22).—See E.P. 200,036; B., 1923, 863.

Making metallo-organic compounds. C. A. Kraus and C. C. Callis, Assrs. to Standard Development Co. (U.S.P. 1,612,131, 28.12.6. Appl., 4.8.23).—See E.P. 214,221; B., 1925, 827.

Manufacture of sugar derivatives of 3 : 3′-diamino-4 : 4′-dihydroxyarsenobenzene. L. Anderson, Assr. to Boots Pure Drug Co., Ltd. (U.S.P. 1,612,598, 28.12.26. Appl., 18.2.21. Conv., 7.1.21).—See E.P. 177, 283; B., 1922, 438.

Manufacture of 1 : 4 : 5 : 8-naphthalenetetracarboxylic acid. W. Eckert, Assr. to Grasselli Dyestuffs Corp. (U.S.P. 1,612,103, 28.12.26. Appl., 31.8.25).—See E.P. 240,859; B., 1926, 7.

Obtaining liquids in a solid state. K. Rast, Assr. to I. G. Farbenind. A.-G. (U.S.P. 1,612,744, 28.12.26. Appl., 15.1.26. Conv., 11.12.24).—See E.P. 259,431; B., 1926, 1029.

Production of hydrated olefines. O. Johannsen and O. Gross (U.S.P. 1,607,459, 16.11.26. Appl., 2.12.25. Conv., 23.8.24).—See E.P. 238,900; B., 1925, 901.

Process for the manufacture of tartaric acid. G. Zemplén, Assr. to Chem. Fabr. Dr. H. Stoltzenberg (U.S.P. 1,605,419, 2.11.26. Appl., 11.12.24. Conv., 5.11.24).—See E.P. 242,590; B., 1926, 995.

***p*-Dialkylaminoarylphosphinous acids.** L. Benda and W. Schmidt, Assrs. to I. G. Farbenind. A.-G. (U.S.P. 1,607,113, 16.11.26. Appl., 26.4.23. Conv., 24.5.22).—See E.P. 214,836; B., 1924, 615.

Process for the production of camphor from *iso*borneol. H. Gammay (U.S.P. 1,607,453, 16.11.26. Appl., 28.1.26).—See E.P. 258,462; B., 1926, 995.

Manufacture of a symmetrical urea [carbamide] of *m*-aminobenzoyl-*m*-aminomethylbenzoyl-1-naphthylamino-4 : 6 : 8-trisulphonate of sodium. E. Fourneau and J. Trefouël, Assrs. to Etab. Poulenc Frères (U.S.P. 1,606,624, 9.11.26. Appl., 26.12.23. Conv., 14.11.23).—See E.P. 224,849; B., 1925, 473.

Process of preparing a measles vaccine and a serum obtained therefrom. R. Degkwitz, Assr. to E. Greutert & Cie. (U.S.P. 1,607,447, 16.11.26. Appl., 6.5.22. Conv., 10.5.21).—See E.P. 179,944; B., 1923, 950 a.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Colour-sensitising photographic plates by bathing. M. L. Dundon (Brit. J. Phot., 1926, **73**, 748—751).—Practical details of sensitising by bathing are given, and recommended procedure is described for sensitising with Acridine Orange, Erythrosin, Erythrosin Silver, Rhodamine B, Pinaflavol, Pinachrome, Pinaverdol, Orthochrome T, Pinacyanol, Pinachrome-Violet, Pantochrome, Napthocyanol, Dicyanine A, Kryptocyanine, and Neocyanine. Wedge spectra of plates bathed according to the advised procedure are presented. W. Clark.

Photographic inversion by heat. H. Belliot (Compt. rend., 1926, **183**, 1279—1280).—Portions of two similar photographic plates were exposed to red and infra-red radiations. One of them was then maintained at 95—100° for 8 hrs., and portions of the exposed parts of both were re-exposed. After development, density measurements showed that the density of the region initially exposed had been lowered by the action of heat, whilst that of the unexposed region was increased. The former region had partly recovered some of its sensitivity for the second exposure, since on the heated plate a higher density was obtained than that resulting from the two successive exposures on the same plate. At higher temperatures the phenomena are produced after a shorter duration of heating. J. Grant.

[Photographic] ripening process. VI. Lüppo-Cramer (Z. wiss. Phot. 1926, **24**, 291—298).—An ammonia-free positive emulsion containing 3% of silver iodide, and prepared from cadmium bromide, had about 1/80 of the sensitivity of a corresponding emulsion prepared from potassium bromide. The emulsion prepared with cadmium salts had an exceedingly fine grain, due apparently to soluble complex formation between the cadmium salts and the silver bromide. In the case of " grainless " emulsions no such difference was found. In this case, however, a large excess of bromide was present, and all the silver was added at once. It is concluded that the differences in emulsions caused by using different bromide salts are due, in the main, to the different solubility of silver bromide in the different bromides. Silver sulphate gives different results from silver nitrate; with certain highly concentrated emulsions silver sulphate caused coagulation of the gelatin. Emulsions prepared with pure sodium bromide were identical with those made from the corresponding amount of potassium bromide. Barium, strontium, and magnesium bromides gave no marked differences. Zinc bromide behaves like cadmium bromide. If lead salts are added to the silver nitrate in the manufacture of an emulsion, insensitive, very fine-grained emulsions result. The influence of inclusions in the crystal lattice and the effect of iodide are considered. W. Clark.

Studies on the Herschel effect. W. Leszynski (Z. wiss. Phot., 1926, **24**, 275—291).—Using ultra-red light of suitable wave-length, the following results were found:—Blackening of a plate caused by exposure to active light, X-rays, α-rays, or hydrogen peroxide is decreased by subsequent exposure to ultra-red light. This occurs both with development before and after fixation, but with silver bromide emulsions no decrease of direct blackening (print-out) was observed. The degree of " lightening " is independent of the wave-length of the first exposure light, and, with constant *It* it is independent of the intensity of the ultra-red exposure over an intensity range of 1 : 75. The " lightening " is, on the other hand, independent of the intensity of the first exposure. However, effects produced by extremely low intensities are not decreased. The sensitivity of a plate to " lightening " decreases with increasing time of exposure to the ultra-red light. A plate bearing a latent image is not affected as regards its sensitivity to active light by exposure to ultra-red light. The sensitivity of a plate not bearing a latent image is, however, slightly decreased by exposure to ultra-red light. The influence of bromide ions and dyes, observed by earlier investigators, is regarded as due to a desensitising action towards active wave-lengths not filtered out from the red light used. An observation of interest in connexion with the Schwarzschild rule is that a plate pre-exposed for 16 sec. to a given density is less sensitive to subsequent exposure than a plate exposed to such an intensity as to give the same density in 1 sec. The results obtained cast doubt on the explanation that the Herschel effect is an oxidation effect, accompanied by a decrease in the amount of silver, although the oxidation explanation can be regarded as a working hypothesis explaining the effect as a dispersion effect with unaltered total silver. Other effects analogous to the Herschel effect are considered. W. Clark.

Electron configuration and light sensitivity. A. Steigmann (Phot. Ind., 1926, 1324—1326).—The paper is mainly a discussion of Fajans' deformation theory, and certain new points of view are brought out in its bearing on the theory of sensitivity. W. Clark.

Patents.

Photography. Technicolor Motion Picture Corp., and E. A. Weaver (E.P. 263,650, 8.2.26).—In stained-gelatin relief images, the image has at least as much density contrast in the shadows as in the half-tones for the same range of intensities in the subject, the upper part of the characteristic curve is concave upwards, and the relief image has a greater thickness contrast in the thicker parts than in the thinner. The image is formed by colouring the sensitised emulsion and printing with light, one component of which is largely absorbed by this colour, and another component of which is largely transmitted by it. The absorption of the emulsion is such as to reduce the first component to a low value, while permitting the other component to penetrate deeply. A greenish-yellow dye is used in the maximum quantity which the film will retain without its crystallising out, and a restrainer is added to restrain such crystallisation. The component of light which is absorbed has a higher actinic power than that which is mainly transmitted. W. Clark.

Production of photographs on paper, parchment, and the like. E. E. Jelley (U.S.P. 1,610,788, 14.12.26. Appl., 1.8.25).—See E.P. 253,380; B., 1926, 693.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 18, 1927.

I.—GENERAL; PLANT; MACHINERY.

Rubber as lining for grinding mills. B. W. Rogers (Ind. Eng. Chem., 1927, 19, 139—141).—The rubber lining to grinding mills raises their efficiency by reducing the power consumption and increasing the fineness of the product. A photographic study of a laboratory mill with a glass front showed that slippage of the grinding media against the shell is detrimental to high efficiency. Slippage is less pronounced in dry-grinding operations than in wet, a corrugated lining helps to prevent it, and a smooth rubber lining prevents it to a greater extent than does smooth steel. A formula has been evolved by which the critical speeds of various diameters and varying pebble charges can be calculated, slippage being ignored; slippage has the effect of reducing the speed below the critical point. Undesirable slippage is minimised as the volume of the grinding charge approaches 50% of the total volume of the mill. The Vulcalock process used for lining makes the mill water- and acid-tight and also protects it from corrosion and abrasion. The rubber lining is primarily adapted for fine wet-grinding mills; in dry grinding its use is limited by the heat factor. Oil and grease have a deleterious effect on rubber, which precludes its use for grinding pigments in oil. It has been found very useful in the ceramic industry, the wear being so slight that no contamination results. M. E. Nottage.

Patents.

Bringing air or other gases to a desired degree of humidity. J. Obermiller (G.P. 432,932, 30.1.24. Addn. to 396,296).—Highly concentrated solutions, after use for regulating the degree of humidity of air or other gases, are brought back to the original strength by the addition of the solid material or by evaporation, or by the addition of water, according to whether they have been used for absorbing moisture from, or adding it to, the gas (cf. B., 1924, 542). L. A. Coles.

Process of carrying on catalytic reactions. F. A. Canon and C. E. Andrews, Assrs. to Selden Co. (U.S.P. 1,614,185, 11.1.27. Appl., 20.6.22).—An exothermic reaction is carried on in a heated gas in the presence of a heated catalyst, and the temperature is maintained approximately at 400—550° by transmitting the excess heat to an alloy of cadmium and mercury which boils at the required temperature. B. M. Venables.

Colour estimating apparatus. Tintometer Ltd., and F. E. Lovibond (E.P. 263,924, 3.10.25).—A white reflecting background, arranged at a predetermined angle (45°) to the line of vision, is viewed simultaneously through the transparent or translucent material to be tested and through one or more transparent standard colour screens. The background comprises a tray containing a white powder, such as precipitated calcium sulphate, providing a flat surface covered by a polished glass sheet of standard colour and thickness, and is illuminated by light of standard colour and intensity from an electric lamp without direct reflection from the glass to the observer's eye. The lamp is mounted within a cylindrical shield. H. Holmes.

Rotary kilns and furnaces. J. H. Bentley (E.P. 263,630, 8.1.26).—A rotary cylindrical kiln is enlarged at one or more places into a conical shape, with the base of the cone towards the upper end of the kiln. The angle between the axis of the cone and its side is greater than the inclination of the kiln to the horizontal. The smaller end of the cone may be equal to or greater than the cylindrical part of the kiln; in the latter case internal lifters are provided to lift the material from the conical to the cylindrical part. Other lifters oppositely inclined may be provided in the conical part to increase the delay of the material there. B. M. Venables.

Furnace. C. B. Dann and G. W. Conduit (E.P. 263,704, 28.8.26).—To ignite unconsumed gases escaping from the furnace a mixture of air and steam is introduced directly into the combustion space without passing over the grate. A hollow vertical partition, formed in sections and closed at the top by removable cover pieces, is provided between the grate portion and the combustion space. The air passes upwards through the partition and through openings in the cover pieces, and the steam is projected through these openings from a swivelling pipe mounted within the partition. The cover pieces may be arched to accommodate the pipe or may be sharply inclined to prevent lodging of fuel on them. H. Holmes.

Furnace. F. A. Lippert (U.S.P. 1,613,615, 11.1.27. Appl., 9.6.24).—A combustion chamber is formed of gradually increasing cross-section away from the inlet (trumpet shaped), and to it is attached a heat-transferring chamber which decreases in section towards the outlet. B. M. Venables.

Heat interchange apparatus. C. F. Higgins (E.P. 263,585, 4.11.25).—The tubes of a heat exchanger are divided into groups, so that the inner fluid passes in zig-zag fashion through each group in succession. The outer fluid passes generally counter-current to the inner fluid, longitudinal divisions being provided so that the groups of tubes for inner and outer fluids are similar. In addition, several transverse baffles are provided with communicating ports so arranged that the outer fluid, when between any one pair of longitudinal divisions, passes zig-zag several times across the tubes. B. M. Venables.

Process and apparatus for refrigeration. T. B. SLATE (E.P. 240,485, 25.9.25. Conv., 27.9.24).—A refrigerator transport car constructed with containers for carbon dioxide ice in the upper part is described.
B. M. VENABLES.

Method of refrigeration. N. B. WALES (U.S.P. 1,613,687, 11.1.27. Appl., 23.4.26).—A volatile refrigerant is compressed and condensed and then volatilised in the presence of a non-absorbent lubricant, which then serves as a brine or cold-transmitting fluid. On its return to the compressor the refrigerant is allowed to entrain a predetermined amount of lubricant for use as such.
B. M. VENABLES.

Refrigerator condenser. C. L. OLIN (U.S.P. 1,613,717, 11.1.27. Appl., 7.8.23. Renewed 18.9.26).—The refrigerant mixed with lubricant is sprayed through holes in a vertical pipe on to cooling coils, and the condensed liquors collect in the lower part of the vessel. The refrigerant is drawn off from the bottom of the vessel and the lubricant from a higher outlet pipe, which is taken down inside the tank to the lowest part before emerging, and is provided with a baffle at its upper or inlet end.
B. M. VENABLES.

Pulverising mills. INTERNAT. COMBUSTION ENGINEERING CORP. (E.P. 251,295, 24.4.26. Conv., 24.4.25).—A feed-regulating device for a grinding mill in which the material is air-borne is arranged so that when the mill partly chokes due to excess feed the vacuum at the outlet will increase and operate a diaphragm which, in turn, operates an electrical switch. The electric current disengages a pawl in the actual feed mechanism.
B. M. VENABLES.

Separating machines. G. PLATH (E.P. 263,660, 23.3.26).—Two forms of combined disintegrating and air-separating machines are described, in each of which the distributing plate (or saturator) which diffuses the solid material into the air stream is formed by the rotating part of the disintegrator. B. M. VENABLES.

Rotary drums for drying materials. J. CUYPERS (E.P. 255,492, 19.7.26. Conv., 20.7.25).—A rotary drying drum is filled with longitudinal shelf-like members, the section of each of which is a three-armed fylfot with up-turned edge extending in the direction of rotation of the drum. Elaborate calculations are made to prove that the necessary speed of rotation of the drum is much reduced and the drying capacity much increased. B. M. VENABLES.

Measurement of the specific gravity of liquid and solid mixtures. T. M. CHANCE (U.S.P. 1,605,171, 2.11.26. Appl., 26.7.24).—To measure the sp. gr. of suspensions of heavier, insoluble, finely-divided solids in a liquid, two pressure gauges are immersed at different depths. The difference in pressure at the two depths is divided by the weight of a column of solid-free liquid equal in height to the difference in depth. H. MOORE.

Filtering apparatus. E. W. W. KEENE (E.P. 261,140, 11.9.25).—The cloth or other filtering medium is held on the surface of the drum of a rotary filter by parallel, equidistant, readily removable strands of wire stretched diagonally across the drum and secured in position. The medium is thus protected from direct contact with the scraper, and is held firmly when subjected to pressure for releasing the cake. A separate straining and tightening device is provided for each strand. H. HOLMES.

[Dual] filters. J. ZWICKY (E.P. 258,905, 27.9.26. Conv., 26.9.25).—Two filters or strainers are combined with a cylindrical valve with six ports so that the oil or other fluid to be strained can flow through either filter or direct from inlet to outlet. B. M. VENABLES.

Apparatus for crystallising liquids. G. T. WALKER (U.S.P. 1,615,151, 18.1.27. Appl., 28.11.21).—The heated liquid is introduced into one end of a long chamber and is discharged through an outlet near the top of the other end. The chamber is provided with means for impelling the liquid through it and with a jacket for the circulation of cooling water. H. HOLMES.

Agitating fluid-separating medium. T. M. CHANCE (U.S.P. 1,614,876, 18.1.27. Appl., 17.5.23).—A fluid mass comprising solids suspended in a liquid lighter than the solids is kept in agitation by a stream of liquid introduced below the centre of the vessel, and deflected by baffles so that when approaching the top the upward velocity is substantially uniform over the whole section.
B. M. VENABLES.

De-aeration of liquids. K. J. SVENSSON and K. A. P. NORLING (E.P. 264,079, 31.7.26).—A liquid such as transformer oil leaving a centrifugal cleaner is freed from small air bubbles by passing it sinuously up and down in a vessel and heating it during one of the upward passes. The partition between the heated pass and the adjacent downward pass is of conducting material so that the liquid is preheated in the latter.
B. M. VENABLES.

Process and apparatus for expressing liquid constituents from materials. F. KRUPP GRUSONWERK A.-G., Assees. of NOBLEE & THORL, NACHFOLG. (E.P. 248,372, 22.2.26. Conv., 25.2.25).—The material falls by gravity into the annular space between two vertical, cylindrical screens, and is pressed and kneaded therein by rotating fingers or wipers, and the pressed material is simultaneously removed from the bottom by upwardly extending, rotating fingers acting as scrapers. Both sets of fingers may be adjustable axially in relation to the screen cylinders to regulate the rate of treatment and the pressure. The rotation and adjustment are relative, *i.e.*, either the fingers or the pair of screens may be the rotating or adjustable part. B. M. VENABLES.

Distillation apparatus. E. R. HAMILTON (U.S.P. 1,614,791, 18.1.27. Appl., 1.11.23).—A retort is divided into a series of chambers on its passage through which the material is heated progressively. An outlet for distillate is provided and, from the lower part of the outer chamber, an outlet for residuum which divides into a valved low-level branch for intermittent use, and an upwardly extending branch for continuous use.
B. M. VENABLES.

Generation of pressure in fire-extinguishers. MINIMAX G.M.B.H. (G.P. 431,554, 27.4.23).—The fire-extinguishers are charged with water and substances which yield with it relatively large volumes of gas, such as alkali or alkaline-earth metals, *e.g.*, sodium or alloys of it with lead, or activated aluminium powder, or compounds of the alkali or alkaline-earth metals with

hydrogen, *e.g.*, calcium hydride, or compounds of these metals with oxygen, with or without the addition of catalysts. For example, sodium peroxide may be used in conjunction with nickel or cobalt sulphate or oxide as catalyst. L. A. Coles.

Absorption method and apparatus. E. Altenkirch, Assr. to Siemens-Schuckertwerke Ges.m.b.H. (U.S.P. 1,615,353, 25.1.27. Appl., 7.2.25. Conv., 14.2.24).—See E.P. 229,332; B., 1926, 519.

Dehydrating unit. P. S. Moyer, Assr. to Aridor Co. (U.S.P. 1,614,753, 18.1.27. Appl., 9.7.23).—See E.P. 205,173; B., 1923, 1243.

Refrigerating apparatus of the absorption type. E. Altenkirch, Assr. to Siemens-Schuckertwerke G.m.b.H. (Re-issue 16,525, 11.1.27, of U.S.P. 1,455,701, 14.8.22. Conv., 8.5.24).—See B., 1923, 638 a.

Pneumatic conveyance of dust, powders, granular materials, etc. J. A. Yates, and Pneumatic Conveyance & Extraction Ltd. (E.P. 264,015, 14.1.26).

[Ash-cooling screens for] furnaces. Babcock & Wilcox, Ltd. From Babcock & Wilcox Dampfkesselwerke A.-G. (E.P. 264,097—8, 24.9.26).

Refrigerator. B. C. von Platen and C. G. Munters, Assrs. to Electrolux Servel Corp. (U.S.P. 1,613,627—9, 11.1.27. Appl., [a] 21.11.25. Renewed 5.11.26. [b] 5.6.26. [c] 11.8.26. Conv., 3.5.24).

[Parallel-motion doors for] annealing and other furnaces, muffles, and the like. H. Wiggin & Co., Ltd., and A. G. Lobley (E.P. 263,916 and 264,114, 1.10.25).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Low-temperature semi-coke in briquetted form. C. V. McIntire and L. R. Thomson (Ind. Eng. Chem., 1927, **19**, 12—15).—An account is given of the plant of the Consolidation Coal Products Co. at Fairmont, W.Va. The coal is carbonised in a McIntire retort, which consists of a horizontal steel cylinder, 8 ft. in diam. and 15 ft. long, having a central shaft provided with radial paddles which agitate the coal during carbonisation, and at the same time propel the mass towards the discharge end of the retort. The semi-coke is ground, mixed with about 11% of a pitch binder, and briquetted. The low-temperature tar supplies sufficient pitch for briquetting requirements. The briquettes are further carbonised in a secondary oven in which a layer two briquettes thick is submitted to radiant heat for 30 min. The by-products from the secondary carbonisation are 2000 cub. ft. of gas (of 450 B.Th.U.) and about 8 gals. of tar per ton of briquettes. The plant has been in operation for about 9 months with an average throughput of 30 tons per day. The final product forms a free-burning fuel suitable for domestic purposes, and sufficiently hard to suffer no deterioration in transport. A. B. Manning.

Fractionation analysis of gas from the low-temperature carbonisation of coal. F. E. Frey and W. P. Yant (Ind. Eng. Chem., 1927, **19**, 21—26).—A method of gas analysis based on the liquefaction and fractionation of the hydrocarbon constituents (cf. Shepherd and Porter, B., 1924, 43) has been applied to gas produced by the carbonisation of a non-coking Utah coal in an experimental vertical retort. In one series of experiments the retort was heated internally by means of superheated steam at 500°, 600°, 700°, and 850°, whilst in a second series external heating was used. A gas from the carbonisation of a West Virginia coking coal in the McIntire retort (cf. preceding abstract) was also analysed. As typical of the gases obtained by internal heating may be taken that at 600°, which gave H_2 10·1; CH_4 40·3; C_2H_4 1·9; C_2H_6 7·4; C_3H_6 1·8; C_3H_8 2·7; C_4H_8 1·4; and C_4H_{10} 0·95%. With internal heating the relative proportions of the gases showed little change over the temperature range studied, with the exception of the hydrogen, which increased with rise of temperature of carbonisation. When the retort was heated externally at 500° analysis of the gas gave results comparable with the above; at 850°, however, the values were H_2 55·7; CH_4 15·0; C_2H_4 1·0; C_2H_6 1·32; C_3H_6 0·58; C_3H_8 0·46; C_4H_8 0·37; C_4H_{10} 0·17%. The proportion of olefines in the total gaseous hydrocarbons, excluding methane, is increased by more drastic conditions of carbonisation. The gas from the McIntire process gave almost the same values on analysis as that produced in the steam carbonisations. A. B. Manning.

Low-temperature carbonisation. The position in England. D. Brownlie (Ind. Eng. Chem., 1927, **19**, 39—45).—A brief description is given of thirteen low-temperature carbonisation processes. A. B. Manning.

The screw as a carbonising machine. I. F. Laucks (Ind. Eng. Chem., 1927, **19**, 8—11; cf. B., 1924, 365).—The development of the Green–Laucks retort is described. Early difficulties were due mainly to the coal, when in the plastic stage, adhering to the cooler parts of the retort wall, and were overcome by heating the shaft of the screw. The retort acts as a gentle "dynamic carboniser" ensuring uniform heating and applying considerable pressure to the mass during the plastic stage, thus yielding a strong and dense coke. The maximum temperature attained is 500°, and deterioration of the metal parts is negligible. The power consumption is low. Some thousand tons of the product have been made in the 36-in. apparatus; a large retort, 9 ft. in diam., is in course of construction. A. B. Manning.

Determination of the coking power of coal. J. G. de Voogd (Het Gas, 1926, **46**, 243—248; Chem. Zentr., 1926, II, 1916).—Kreulen's method (cf. Het Gas, 1924, **44**, 321), *i.e.*, admixture of powdered coal with different proportions of sand, and determination of the strength of the coke formed, is unreliable, but fairly consistent results were obtained working on the lines of Meurice's method (cf. B., 1924, 545), the index numbers obtained comparing favourably with values obtained by the crucible test at higher and lower temperatures. It is essential that a freshly powdered test portion of coal be used; storage in the air or in closed bottles, or drying at 105°, lowers the index number considerably. L. A. Coles.

Heat of coking of gas- and coking coals. E. Terres and H. Wolter (Gas- u. Wasserfach, 1927, **70**, —15, 30—34, 53—58, 81—85; cf. Strache and Grau,

B., 1921, 289 A; Davis, Place, and Edeburn, B., 1925, 656).—The heats of coking of six bituminous coals have been determined at coking temperatures between 650° and 1100° in a specially designed calorimeter. The finely-powdered coal (15 g.) was carbonised in an electrically-heated silica retort (26 mm. diam. × 65 mm. high) contained in a bronze bomb immersed in a calorimeter. The volatile products passed down through an outlet tube in the base of the retort directly into a second calorimeter, in which the water and tar were condensed, whilst the gas, after attaining the temperature of the calorimeter, passed out to the atmosphere. The coking temperature was determined by a thermocouple placed at the centre of the coal. In order to attain the high temperatures required in a convenient time efficient heat insulation in the form of a double-walled, clay cylinder was interposed between the retort and bomb walls. The determination was carried out by first coking the coal at the desired temperature, the input of electrical energy, the rate of which was maintained constant throughout all the experiments, being accurately measured. The coke was then allowed to cool, and at the moment that it reached 600° the temperature of the upper calorimeter was recorded. From a series of preliminary experiments, using quartz sand in the retort, curves had been constructed giving the heat content of the retort itself (and its heat insulation) when the sand had cooled to 600°, as a function of the total heat input. From (*a*) the total heat input, (*b*) the heat content of the retort itself, (*c*) the heat that had passed into the upper calorimeter, (*d*) the heat required to raise the temperature of the coke from 600° to the coking temperature, and (*e*) the heat given up by the volatile products, the heat of coking was calculated from $h = a - (b + c) + d + e$. To determine d measurements were made of the sp. heat of the semi-cokes and cokes from one coal ("Präsident") over the required temperature ranges. The sp. heat of the semi-coke is greater the lower the carbonising temperature. The accuracy of the results is estimated to be within $\pm$ 1%, except for coals with a high heat of decomposition (up to 30 g.-cal./g.) when the uncertainty may be $\pm$ 2%. The gas-coal "Teutoburgia" shows a linear increase of heat of coking with temperature, from 308·3 g.-cal./g. at 740° to 467·5 at 1033°. The coking coal "Zollverein" behaves similarly up to 1000°, above which temperature it exhibits abnormally high values. The heats of coking of the three gas-coals "Unser Fritz," "M. Stinnes I/II," "Graf Moltke" have minimum values between 800° and 900°, though at different temperatures for each. The "normal" heat of coking, *i.e.*, the value at 1000°, is nearly the same for all the above coals (400—420 g.-cal./g.). The coking coal "Präsident" is an exception; its heat of coking rises uniformly up to 850°, and thence to 1050° remains constant at 340 g.-cal./g. The occurrence of minima in the heats of coking are possibly due to exothermic reactions taking place during decomposition. On heating the coals in an electric furnace and plotting coal temperature and furnace temperature against time, all the coals with the exception of the "Zollverein" gave evidence of such exothermic reactions. Other factors, therefore, must also be concerned in the production of the minima observed, and the rate of evolution of the volatile matter probably plays some part. The possibility of classifying coals from their heats of coking is discussed, but the greater value of such determinations lies in their practical application to the heat balances of coke ovens and gas retorts.

A. B. MANNING.

Heat of distillation of coal. S. P. BURKE and V. F. PARRY (Ind. Eng. Chem., 1927, **19**, 15—20; cf. preceding abstract).—A charge of coal was carbonised in a current of hot inert gas and the heat content of the gas determined before and after passage through the coal. The retort containing the coal was surrounded by an electrically-heated adiabatic furnace, so that no loss of heat occurred by radiation. The determination was repeated on the empty retort, the data then giving by difference the heat required to carbonise the coal under the conditions of the experiment ("heat of distillation"). The retort, 6 in. in diam. and 20 in. long, held 18 lb. of coal, and the maximum temperature of carbonisation, *i.e.*, the temperature of the gas at entry, was 593°. The gas temperatures, ingoing and outgoing, were measured by chromel–alumel thermocouples, and the sp. heat of the gas, a flue gas containing no oxygen, was calculated from its composition. Corrections were made for minor heat exchanges in the adiabatic furnace. The apparatus was calibrated by filling with copper, and gave a value for the mean sp. heat between 15·5° and 593° of 0·097, the accepted value being 0·099. The heat of distillation of a lignite, a non-coking Utah coal, and a non-coking Pittsburgh coal were, respectively, 477, 408, and 407 B.Th.U./lb. An estimate was made of the heat content of the products of carbonisation, and gave by difference the approximate values 85, 37, and 7 B.Th.U./lb. (exothermic) respectively, for the heats of carbonisation of the above three coals.

A. B. MANNING.

Rate of combustion of coal dust particles. II. Effect of particle size on pressure increase attending inflammation of coal dust. C. M. BOUTON and J. H. HAYNER (Carnegie Inst. Tech., Min. Met. Invest. Bull., 1925, **22**, 1—23; Chem. Abstr., 1926, **20**, 3572).—The explosibility of coal does not increase as the fineness of the dust, particles of diameter 10—25 μ having maximum inflammability.

A. A. ELDRIDGE.

Derivation of coal from lignin. H. STRACHE (Brennstoff-Chem., 1927, **8**, 21—22).—The conversion of lignin into humic acids and anhydrides is well demonstrated by the case of a recently-felled "1000-years old" oak, the centre of which was made up of a reddish-brown, earthy substance, partly soluble in warm water and nearly completely soluble in cold alkalis, and having a carbonyl value about equal to that of a young brown coal. Lignite formation had not occurred, and it is probable that the composition of the wood from which lignite is formed differs quantitatively, as opposed to qualitatively, from that which is the parent of earthy brown coals.

W. T. K. BRAUNHOLTZ.

Brief résumé of the fuel field. S. W. PARR (Ind. Eng. Chem., 1927, **19**, 7—8).—An historical summary of the development of fuel research in America during the last hundred years, and a brief description of the Parr–Layng carbonisation process (cf. B., 1926, 697).

A. B. MANNING.

Calculation of the calorific value of fuels from their chemical composition. R. VONDRACEK (Brennstoff-Chem., 1927, **8**, 22—23).—As a result of some 200 coal analyses, it is found that the cal. value of a fuel is most accurately calculated from the equation: $H_0 = (78 \cdot 6 + 2 \cdot 8 \sqrt{100 - C'})C + 270 (H - 0 \cdot 1\ O) + 25 S$, where C′ is the calculated carbon content of the pure coal. Within the limits C = 90 — 45% this may be written: $H_0 = (89 \cdot 1 - 0 \cdot 062\ C')C + 270 (H - 0 \cdot 1\ O) + 25 S$. The difference between calculated and determined cal. values is generally less than 0·5%, and the formula also gives fairly good results with liquid fuels.
W. T. K. BRAUNHOLTZ.

Calorific value of gas and its determination by means of a calibrated gas holder and Junkers' calorimeter. R. GEIPERT (Gas- u. Wasserfach, 1927, **70**, 15—16).—A determination of the calorific value of a gas of which only 10 litres are available can be made by passing the gas from a calibrated glass gas holder to the calorimeter, the temperature of the current of water in the latter having first been brought approximately to the value expected. The results are of the same degree of accuracy as those obtained by the usual method. A. B. MANNING.

Radiation from luminous flames. R. T. HASLAM and M. W. BOYER (Ind. Eng. Chem., 1927, **19**, 4—6; cf. B., 1925, 388).—The heat radiated from luminous flames of town's gas, methane, ethylene, and acetylene has been determined by allowing the radiation from a small flame to fall on a thermopile at such a distance from the flame that it could be considered as a point source. The radiation from the flames of the first three gases amounted to 9·2, 8·0, and 10·65% respectively of the total heat of combustion. These values, however, are considered to be low, because of the difficulty of obtaining complete luminosity in such a small flame. Acetylene gave a truly luminous flame, the radiation from which amounted to 28·2% of the total heat of combustion, as compared with 6·9% from a similar non-luminous acetylene flame. The results indicate that transference of heat by radiation may be of greater importance than is usually recognised, and attention is directed to Lent's suggestion for increasing the rate of heat transfer from non-luminous flames by inoculating them with an illuminating agent (Wärme, 1926, **49**, 145).
A. B. MANNING.

Elimination and recovery of phenols from crude ammonia liquors. R. M. CRAWFORD (Ind. Eng. Chem., 1927, **19**, 168—169).—The process operated at Troy, N.Y., for the recovery of phenols from crude ammoniacal liquor by extraction with benzene (cf. B., 1926, 348) has been modified as follows. The phenols are extracted from benzene solution by sodium sulphide solution in place of sodium hydroxide; the phenols are then liberated by acidifying with hydrogen sulphide gas. The spent liquor, a mixture of sodium sulphide and sodium hydrosulphide, is boiled with regeneration of sodium sulphide and hydrogen sulphide. The process is thus a cycle, and only slight mechanical losses of soda and hydrogen sulphide have to be made up by new material. C. IRWIN.

Desulphurising action of silica gel [on oils]. H. I. WATERMAN and M. J. VAN TUSSENBROEK (Brennstoff-Chem., 1927, **8**, 20—21; cf. B., 1926, 621).—A solution of ethyl sulphide in benzine (b.p. 40—60°) containing 1% of sulphur was shaken with silica gel of various dimensions and moisture content. The larger-sized gel (0·6—0·7 mm.) removed least sulphur, but practically no difference was found between the action of gel of 0·29—0·37 mm. and that of 0·17—0·20 mm. Increasing the moisture content from 6% to 10% practically destroys the adsorptive action of the gel towards ethyl sulphide. W. T. K. BRAUNHOLTZ.

Composition of light oils from low-temperature carbonisation of Utah coal. R. L. BROWN and R. B. COOPER (Ind. Eng. Chem., 1927, **19**, 26—31).—The coal was distilled in a vertical retort internally heated by means of superheated steam, of which the inlet temperature was 736°. After separation of the tar and liquor, the light oil from the gas was condensed in vessels cooled by solid carbon dioxide and acetone or by liquid air, the yield being 1·7 gals. per ton. The oil boiled from 20° to slightly above 200°, and had a high olefine content. It contained about 30% of amylenes and 10% of pentane (principally *iso*pentane). About 26% consisted of six-carbon-atom compounds, of which about two-thirds were hexenes and one-third saturated compounds (chiefly cyclic). The remainder of the light oil consisted of olefines, naphthenes, and paraffins, the first constituting rather more than half. The amount of acids and bases present was very slight. A. B. MANNING.

Blümner cracking process. D. LOHMANN (Petroleum, 1926, **22**, 1247—1251).—This process operates in liquid–gas phase at a pressure of 40 atm. without catalysts, at a temperature of about 450°. By the high pressure the formation of permanent gases and coke is reduced to a minimum. The preheater, autoclave, and cracking chamber only are under pressure. The oil is pumped in through a preheater where it is warmed to 200°, and through pipes heated by the waste gases, where it attains 330—350°. The autoclave contains molten lead, in which is a basket filled with Raschig rings, and the oil, introduced at the bottom, streams upwards through the filling, not coming into contact with the sides of the vessel. From the autoclave, the oil passes to alternate cracking chambers, one of which may be cleaned from coke while the other is in action. The lead is not acted on by the sulphur compounds of the oil, and may remain in use for a long period. After fractionation, the spirit is easily refined by 1% of sulphuric acid, and is strongly anti-detonating. The process is used with success for cracking lignite oils.
H. MOORE.

"Oxygen value" method for the examination of petroleum products. S. NAMETKIN and L. ABAKUMOVSKY (J. pr. Chem., 1927, **115**, 56—74).—The sources of error in the method for the determination of unsaturated hydrocarbons by means of perbenzoic acid in chloroform solution (Nametkin and Brüssoff, A., 1926, 420) have been investigated, and the method applied to various mineral oils. Consistent and accurate results are obtained when the following conditions are observed: (1) a blank experiment is carried out with each determination owing to the gradual decomposition of the per-

benzoic acid solution; (2) 10 c.c. of a 0·5N-solution of perbenzoic acid in chloroform and not more than 0·3—0·4 g. of material are used for each determination, less material being used if a large proportion of unsaturated substances is present, the amount of these not exceeding the quantity corresponding to one third of the perbenzoic acid used; (3) the oxidation is carried out in the dark at 9—12° for 40—48 hrs.; (4) the perbenzoic acid solution should be prepared 1—2 days before use. By this method is obtained the oxygen value (O.V.) of the substance, corresponding to the iodine value (I.V.), and is defined as the quantity of active oxygen from the perbenzoic acid required for the oxidation of 100 g. of the substance. The ratios 100 O.V./16 and 100 I.V./254 then represent the same value, the "unsaturation coefficient," and in most cases the values obtained by the two methods agree closely. In some cases, however (*e.g.*, Emba kerosene distillate), the O.V. value is larger than that for the I.V., due to the presence of certain constituents which are attacked by perbenzoic acid but not by iodine. These methods are applied to the investigation of various samples of motor spirit, light petroleum, lubricating oils, Emba and Baku oils, in order to determine the proportion of unsaturated compounds present, the results being tabulated and discussed. In general, it is found that the more drastic the acid treatment used in the refining process, the smaller the proportion of unsaturated substances present in the oil. J. W. Baker.

Impregnating wood with paraffin. Eberlein and Burgess.—See IX.

Patents.

Gasification of fuel. G. Szikla and A. Rozinek (E.P. 255,857, 13.7.26. Conv., 25.7.25).—Coarse coal dust is fed to the bottom of a shaft where it is carried upwards by an air blast. The amount of air supplied is sufficient only to effect the producer-gas reaction, which is brought about by the elevated temperature in the shaft. The resulting gases may carry unburned coal particles along with them. These particles are entrapped by suitable baffles and returned to the base of the shaft. R. A. A. Taylor.

Low-temperature carbonisation of coal, lignite, shale, peat, or the like. H. Whitehead and H. P. Hird (E.P. 263,686, 8.7.26).—A retort is made of cold-blast cast-iron and facilities are provided for having in it one or more vertically-placed bars made to revolve. The bars may be straight or twisted, or these two forms may be interchangeable. They serve to compress the charge while it is being coked and to provide a central passage for the products of distillation. R. A. A. Taylor.

Improving catalytic processes. E. A. Prudhomme (F.P. 606,897, 17.2.25).—Processes such as the production of liquid hydrocarbons from lignite are improved by the introduction at different stages of gases capable of increasing the proportion of atomic hydrogen, carbon monoxide, or other highly active gases. L. A. Coles.

Purification of [desulphurising] industrial gases, and production of carbonyl sulphide. Soc. Nationale de Recherches sur le Traitement des Combustibles (F.P. 607,296, 16.3.25).—Gases containing sulphur compounds are scrubbed with alcoholic sodium hydroxide and the solution obtained, which contains sulphur and carbonyl sulphide, is treated with dilute mineral acid. L. A. Coles.

Method and apparatus for charging and discharging trays used in dry distillation of bituminous materials. Patentaktiebolaget Gröndal-Ramén (E.P. 250,227, 27.3.26. Conv., 6.4.25).—The bituminous material is charged to the retort in truck-containers which consist of a series of superposed trays. The truck has a horizontal cross axle on which it can be turned up so that the fore end comes under a feeding device. When filled, each tray is isolated by a cover. The discharging device is similar, but a scraper is provided which passes over the bottom of each tray to remove any adhering coke. R. A. A. Taylor.

Destructive distillation of bituminous materials. C. N. Forrest and H. P. Hayden (E.P. 262,959, 28.12.25).—Bituminous materials of liquid or semi-fluid character are gasified and cracked by this process in a suitable form of producer with the necessary condensing, scrubbing, and collecting plant for the products. A refractory material of suitable size is impregnated with the bituminous substance, which is thus fed to the producer. After the cracking process the refractory material may have a coke deposit accompanying it; it is taken out at the base of the producer, the coke is burnt off, and the carrier used again. R. A. A. Taylor.

Oil still. J. E. Bell, Assr. to Sinclair Refining Co. (U.S.P. 1,613,306, 4.1.27. Appl., 22.1.25).—Hot gases from the firebox pass through tubes immersed in the oil, and then return through two central flues through the still, thence to the stack; by-passes are made between the firebox and the flue adjacent to the stack. There are air-heating passages in the central flues, the air inlet being at the firebox end of the still, and the discharge at the opposite end. W. N. Hoyte.

Heating distillation chambers. W. Drähne (G.P. 433,849, 25.3.25).—Retarding devices are provided at the charging end of a relatively long distillation chamber heated by intermittent firing, but not at the outlet end, so that the heat is suitably distributed throughout the charge. L. A. Coles.

Continuous distillation of petroleum oils. F. A. Howard, Assr. to Standard Development Co. (U.S.P. 1,606,075, 9.11.26. Appl., 1.9.21).—Water-free oil is passed in a stream through a heated zone, and rapidly brought to a temperature at which certain of its constituents are normally vapours. An inert gaseous material is introduced into the stream, which discharges into a chamber at a higher level. The incoming oil is heated, and travels through a tortuous path in the vapour space of the chamber before entering the body of oil. Vapours evolved leave the chamber in countercurrent to the entering oil. H. Moore.

Process and apparatus for the purification [desulphurisation] and hydrogenation of liquid hydrocarbons. C. D. Maze (E.P. 263,381, 27.4.26).—A mixture of steam and vaporised or atomised oil is projected on to a heated catalyst; the formation of nascent hydrogen leads to desulphurisation and hydro-

genation. The catalysts mentioned are iron, nickel, copper, cobalt, zinc, cadmium, vanadium, tungsten, and carbon at 350—450°, and the proportion of steam to hydrocarbon 2 : 1. All catalysts except the last-mentioned may be regenerated by increasing the proportion of steam present in the mixture. W. N. HOYTE.

Revivification of fuller's earth. L. A. TARBOX, Assr. to EMLENTON REFINING Co. (U.S.P. 1,613,299, 4.1.27. Appl., 10.4.25).—The spent earth is drawn in a stream through a furnace, where its temperature is steadily raised out of contact with the flue gases. Air is introduced at various points in the stream in sufficient quantity to burn off the carbonaceous matter. W. N. HOYTE.

Production of montan wax from lignite. A. RIEBECK'SCHE MONTANWERKE A.-G. (G.P. 432,744, 17.8.23. Addn. to G.P. 325,165; B., 1920, 814 A).—Either the benzene or toluene in the earlier process is replaced by the higher-boiling fractions of high- or low-temperature coal or lignite tar, or of petroleum, having a b.p. above 150°, or the mixtures of low-boiling alcohols and acetone are replaced by higher-boiling (*i.e.*, about 140°) oxygenated solvents (alcohols or ketones), or both lower-boiling constituents are replaced by the higher-boiling substances. The accumulation of pressure during extraction is avoided. R. A. A. TAYLOR.

Production of montan wax. O. L. BEER (G.P. 433,364, 18.1.24).—Starting with decaying vegetable matter, the bitumens in the residues insoluble in alkali are increased by heating the mixture above the b.p. of the products of reaction, the requisite quantity, but no excess, of alkali having previously been added to combine with the humic acids. R. A. A. TAYLOR.

Avoiding shock chill in chilling and separating substances. H. A. GILL. From SHARPLES SPECIALTY Co. (E.P. 261,450, 20.7.25).—In separating wax from petroleum oils or stearine from vegetable oils, the liquid is gradually cooled by circulating brine coils from a refrigerating plant, so that the refrigerant never differs in temperature from the liquid by more than a small fraction of the total range through which the liquid is cooled. The wax is separated as a discontinuous precipitate, and its subsidence is prevented by agitation. In this form it is more coarsely divided than when shock chilled, contains less occluded oil, and is easily separated by centrifuging. The temperature of the brine in the circulating pipes is adjusted by small additions of colder brine, to maintain a temperature difference of 2—3° between the cooling medium and the oil. H. MOORE.

Petroleum-treating apparatus. J. W. LEWIS, Assr. to ATLANTIC REFINING Co. (U.S.P. 1,606,248, 9.11.26. Appl., 3.4.19).—Horizontal drums are placed at different levels in a heating chamber in a pressure-still structure. A still outside the chamber extends from above to below the drums. The drums and still are connected, and there is a vapour discharge line above the liquid level in the still. H. MOORE.

Conversion of heavy hydrocarbon oils into lighter hydrocarbon oils. H. T. WRIGHT and F. ESLING (E.P. 261,077, 10.8.25).—Oil is preheated in a retort or pipe still to a temperature at which it would vaporise under the pressure obtaining in the vaporiser, but without vaporising or cracking. Thence it passes to a vaporiser, and further to a cracking chamber, a dephlegmator, fractionating chamber, and condenser. In the vaporiser a portion of the oil is vaporised, the rest being returned for retreatment, the cracking temperature being confined to that effective for the fractions vaporised. Certain of the fractions are withdrawn for subsequent treatment. The retort has a continuous tube of small bore, through which the oil travels at a high velocity from the coolest to the hottest part. The vaporiser is a vertical cylinder heated by the furnace gases, and the oil runs down its inner vertical walls. Deposited asphalt and tar may be blown out in the form of sludge. The oil vapour is submitted to cracking temperature in the cracking chamber, the heating surface consisting of small-bore zig-zag tubes through which the oil travels at high velocity. The vapour expands into the expansion chamber, where the heavier portions are condensed, and passes to the dephlegmator and fractionating chamber, where the kerosene is cut from the petrol, which is condensed in the condenser. In the process the original oil is separated into several groups of different boiling range, one group being returned for retreatment, while the others are stored for subsequent use. H. MOORE.

Apparatus for cracking oil. G. L. PRICHARD, Assr. to GULF REFINING Co. (U.S.P. 1,605,063, 2.11.26. Appl., 20.11.20. Renewed 18.9.26).—Oil is treated under pressure in a still containing a stirrer driven by a shaft, made tight by stuffing boxes and passing through a housing chamber containing cooled fluid under pressure. H. MOORE.

Absorption tower for hydrocarbons. J. E. KOBERNIK, Assr. to NEWTON PROCESS MANUF. Co. (U.S.P. 1,613,352, 4.1.27. Appl., 1.4.25).—The tower is divided into sections by horizontal partitions, and the gas to be treated with absorption medium passes upwards through a slot in each partition, the sides of which extend upwards. A cap is situated over and round each slot deflecting the gas downwards; it then passes through perforations in a horizontal partition situated a short distance above the main partition; the absorption medium is maintained above the perforated partition, the depth of the medium being limited by overflow pipes conveying it from one section to the next lower one. W. N. HOYTE.

Production of residual oil products. E. E. BARTELS, Assr. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,613,737, 11.1.27. Appl., 17.11.24).—Continuous distillation is carried out in a battery of stills, the residue from an intermediate still being withdrawn and blended in ascertained proportions with the residue from the final still to give the desired product. W. N. HOYTE.

Motor fuel. A. B. HAMBY and J. D. WOODWARD, Asssrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,606,431, 9.11.27. Appl., 4.1.22).—The fuel comprises gasoline principally, aniline, and motor benzol. H. MOORE.

Manufacture of non-emulsifiable mineral oil of high dielectric strength. H. T. MAITLAND, Assr. to SUN OIL Co. (U.S.P. 1,605,046, 2.11.26. Appl., 24.3.23). —A distillate of crude petroleum is treated with alkali to saponify contained fatty acids and the soaps are

separated, suspended matter being washed out of the distillate by steam. The distillate is treated with sulphuric acid and the sludge separated, then treated with alkali and the precipitate separated, and again washed with steam. After an alkali treatment fuller's earth is admixed and the distillate reduced.

H. Moore.

Production of emulsions of lubricating oils. Oelwerke Stern-Sonneborn A.-G. (G.P. 432,683, 19.7.22. Addn. to 429,551).—The tar distillates of the earlier process are partly or wholly replaced by oils or oil mixtures which are themselves other than lubricating oils. These are converted into stable emulsions by means of lime-water, certain highly-viscous, emulsifying lubricants being added. R. A. A. Taylor.

Separation of the constituents of [mineral oil] emulsions. J. H. Brégeat (F.P. 608,154, 26.3.25).—The emulsion is methodically and continually washed with liquid phenols, their hydrogenated derivatives, naphthols, aromatic sulphur compounds, or mixtures of these. Pressure may be used. R. A. A. Taylor.

Apparatus for the purification and dehydration of fats, oils, and hydrocarbons. Apparatebau A.-G. Bühring (G.P. 433,159, 12.5.25).—The oil to be purified is pumped out of the container to and fro through the purifier, which consists of a filter and vaporiser. The latter is surrounded by a heating fluid which is kept at a higher temperature than that of the water boiling under reduced pressure. The vapours are thus superheated and collect outside the vaporiser in a special condenser, in which they may be measured so that the water content of the impure oil can be determined. Transformer oil is thus rendered suitable for high-voltage processes. R. A. A. Taylor.

Production of neutral bitumens by means of acid resins from the purification of mineral oil. C. Sautermeister and F. Wilhelm (F.P. 606,817, 24.11.25. Conv., 30.6.25).—The sulphuric acid contained in the acid resin is poured off and reduced with hydrogen sulphide, a current of the gas being passed into the bitumen or generated in it from sulphur.

R. A. A. Taylor.

Bituminous emulsion. Soc. Rol Lister & Cie. (F.P. 608,123, 21.12.25).—A soap solution is used, prepared from rosin, cottonseed oil, or oleic acid and caustic soda, and emulsified by means of a high-speed turbine. R. A. A. Taylor.

Prevention of corrosion in the manufacture of fuel gases. C. Cooper and D. M. Henshaw, Assrs. to W. C. Holmes & Co., Ltd. (U.S.P. 1,614,347, 11.1.27. Appl., 30.1.26. Conv., 11.12.24).—See E.P. 248,841; B., 1926, 428.

Purifying, enriching, or refining crude graphite. E. J. E. Dumond (U.S.P. 1,614,352, 11.1.27. Appl., 8.4.25. Conv., 23.4.24).—See E.P. 232,936; B., 1925, 803.

Separating mineral oils or the like from sand or rock. M. E. Fyleman (U.S.P. 1,615,121, 18.1.27. Appl., 16.11.20. Conv., 3.3.20).—See E.P. 163,519; B., 1921, 460.

Recovery of volatile substances. J. Jannek, G. Wietzel, and F. Stoewener, Assrs. to I. G. Farbenind. A.-G. (U.S.P. 1,614,615, 18.1.27. Appl., 17.8.25. Conv. 5.9.24).—See F.P. 604,207; B., 1926, 776.

Method of refining hydrocarbons. O. D. Lucas and E. L. Lomax, Assrs. to V. L. Oil Processes, Ltd. (U.S.P. 1,615,286, 25.1.27. Appl., 30.4.24. Conv., 14.11.23).—See E.P. 223,066; B., 1924, 1006.

Art of pyrogenetic conversion of hydrocarbon oils. F. A. Howard, E. M. Clark, and J. R. Carringer, Assrs. to Standard Development Co. (U.S.P. 1,615,384, 25.1.27. Appl., 23.5.21. Conv., 15.1.21).—See E.P. 174,089; B., 1923, 593 a.

Gaseous or powdered fuel burner. J. D. Troup (E.P. 263,710, 16.9.26).

Colour of oils (E.P. 263,924).—See I.

Distillation apparatus (U.S.P. 1,614,791).—See I.

De-aeration of transformer oil (E.P. 264,079).—See I.

Distillation of petroleum (U.S.P. 1,614,483).—See III.

Desulphurising gases (F.P. 607259).—See VII.

III.—TAR AND TAR PRODUCTS.

New type of tar. S. R. Church (Ind. Eng. Chem., 1927, **19**, 31—34).—The tars produced from two types of Illinois coal by carbonisation in a Parr–Layng retort at temperatures from 750° to 850° have been examined. They possess the characteristics of a good quality high-temperature tar, their average composition on the basis of a yield of 15 gals. per ton of coal being: light oil (crude benzene), 1·5 gals.; No. 1 creosote, 5·1 gals.; tar acids (rich in cresols), 1·6 gals.; pitch, 6·6 gals.

A. B. Manning.

Low-temperature semi-coke briquettes. McIntire and Thomson.—See II.

Recovery of phenols from crude ammonia liquor. Crawford.—See II.

Patents.

Apparatus for continuous preliminary treatment of crude benzols. Soc. des Établ. Barbet (E.P. 244,130, 7.12.25. Conv., 5.12.24).—The vapours of the crude benzol are fractionated in two rectifiers in series, the first operating at atmospheric pressure, the second under vacuum. The benzol is vaporised in three stages, the three sets of vapours being introduced at appropriate points in the rectifiers. The height of the vacuum rectifier is so arranged that the withdrawn condensate collects at ground level without the use of a pump.

W. N. Hoyte.

Cracking of mixtures of hydrocarbons poor in hydrogen. Braunkohlen-Produkte A.-G. (G.P. 432,851, 1.2.24).—The mixture of tar, asphaltum, etc. is cracked by heat in the presence of hydrocarbons rich in hydrogen so that the greater part of the mixture consists of tar. The vapours of the mixture are heated in the presence of a metallic contact mass, preferably of aluminium, magnesium, or alloys rich in these metals.

R. A. A. Taylor.

Production of lubricating oil from coal-tar oil. ZECHE M. STINNES, Assee. of W. EICKEL (G.P. 432,985, 12.4.25).—The tar is distilled until the residue cokes. The fraction above 270° is separated from any solids it may contain, diluted with an inert, low-boiling solvent, refined first with alkali solution, then with dilute sulphuric acid, the solvent is then driven off, and the residual oil is distilled *in vacuo*. Sulphur dioxide may be used for refining the oil in place of sulphuric acid, but the solvent must not be miscible with the gas. In the vacuum distillation of the oil any solids separating on cooling the fraction 200—270° (at normal pressure 300—370°) are removed. The oils produced are free from asphaltum, and of good quality. R. A. A. TAYLOR.

Purification of beechwood tar oils and their constituents. H. SUIDA and A. WACEK (F.P. 602,866, 1.9.25. Conv., 20.5.25).—The oils, before or after removal of neutral constituents, or fractions of different b.p. derived from the oils, are heated under pressure with concentrated hydrochloric acid. For example, on treating the fraction of b.p. 240—270°, methyl chloride (32 pts. per 100 pts. of oil) is formed, and an oily product remains from which pyrogallol and its *C*-methyl and *C*-propyl derivatives can be isolated. Phenol, creosols, and ethylphenol, as well as pyrocatechol and methylpyrocatechol, are obtained from the fraction of b.p. 199—220°. L. A. COLES.

Production of aromatic hydrocarbons. GELSENKIRCHENER BERGWERKS A.-G. ABTG. SCHALKE (Austr. P. 101,681, 11.4.24).—Neutral oil of b.p. up to about 200° from low-temperature tar is heated at 500—700°, alone or in admixture with non-reacting gases, and in the presence or absence of catalysts, such as palladium, silver, copper, iron, nickel, carbon, or alumina. The product consists mainly of a mixture of aromatic hydrocarbons, but if fractions of the oil of b.p. 76—82°, 105—115°, and 135—145° are treated separately, the products consist mainly of benzene, toluene, and xylenes respectively. L. A. COLES.

Distillation or evaporation of liquids [tar, petroleum]. J. L. MAJOR and B. TAYLOR (U.S.P. 1,614,483, 18.1.27. Appl., 23.1.25. Conv., 7.2.24).—See E.P. 233,419; B., 1925, 536.

Impregnation of roads (E.P. 263,975).—See IX.

Condensation products from tar oils (F.P. 607,655).—See XIII.

Ink from tar extract (G.P. 433,158).—See XIII.

IV.—DYESTUFFS AND INTERMEDIATES.

PATENTS.

Preparation of dyes, colouring matters, and the like [stable, water-soluble, vat-dye derivatives]. B. WYLAM, J. E. G. HARRIS, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 263,898, 5.8.25).—Vat dyes are treated with an alkyl chloroacetate in the presence of a tertiary organic base, such as pyridine, quinoline, or their homologues, and a suitable metal such as zinc or copper. The base may be partially replaced by a neutral diluent. The products, on treatment with warm dilute alkali, yield solutions of derivatives of the vat dyes which may be used for dyeing or printing textile materials as described in E.P. 245,587 and 247,787 (B., 1926, 235, 403). *E.g.*, 10 pts. of flavanthrone are suspended in 70 pts. of pyridine, and 10 pts. of copper and 50 pts. of ethyl chloroacetate are added. The mixture is stirred and heated at 100° for a short time, then poured into water, filtered, and the solid washed free from pyridine etc. The blue-black paste so obtained is heated at 60—80° with 5% sodium hydroxide solution, when a deep blue-violet solution is formed. A. DAVIDSON.

Preparation of dyestuff inks. I. G. FARBENIND. A.-G., Assees. of H. SCHLADEBACH and H. HÄHLE (G.P. 431,369, 14.1.25. Addn. to 430,582).—Formaldehyde or substances yielding it are added to solutions of substantive dyestuffs in the presence or absence of alkali. *E.g.*, about 12 pts. of a dyestuff are dissolved in 950 pts. of boiling distilled water, the cooled and filtered solution is treated with 50 pts. of 30% formaldehyde, and made up to 1000 pts. The ink marks after a short time become completely waterproof. B. FULLMAN.

Azo dyestuffs and process of making same. H. WAGNER, W. LANGBEIN, K. BECK, and K. THIESS, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,613,919, 11.1.27. Appl., 29.1.25. Conv., 8.2.24).—See E.P. 228,913; B., 1925, 839.

Preparation of aminodianthrimides. H. M. BUNBURY, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,616,109, 1.2.27. Appl., 17.10.25. Conv., 19.12.24).—See E.P. 248,866; B., 1926, 398.

Pigments (G.P. 432,216).—See XIII.

Fixing pigments and dyes (G.P. 433,152).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Lignin from pine wood by the action of dry-rot. C. G. SCHWALBE and A. EKENSTAM (Cellulosechem., 1927, 8, 13—15).—The residue which is obtained by the action of dry-rot (*Merulius lachrymans*) on pine wood consists of lignin 73%, cellulose 15%, other carbohydrates 8%, resin 4%, and contains no trace of pentosans. The fats present in the original wood are apparently destroyed by the fungi, and the residual 15% of cellulose is partially decomposed. About 12% of the material is soluble in cold water, but the solution contains no fermentable sugars. On hydrolysis with dilute hydrochloric acid at 100°, however, the water extract yields considerable quantities of sugars which are partially fermentable. The lignin isolated from the residue by means of hydrochloric acid differs from lignin from fresh wood prepared by the same method in having a lower methoxyl content (10·2%), and in being soluble to the extent of 64·2% in 5% sodium hydroxide solution. In the isolation of lignin by Willstätter's method from both fresh and rotten wood, the residual lignin is liable to contain cellulose, owing to the rapid swelling action of the highly concentrated acid used and the consequent prevention of diffusion of the acid to the interior of the wood. The difficulty may be overcome by treating the wood first with relatively dilute acid (25%) and gradually increasing the concentration of acid to 42%. Under these conditions the lignin is more rapidly and completely freed from cellulose, and the product obtained is more easily filtered.
W. J. POWELL.

Cellulose from nettles. O. Routala and J. Sevón (Cellulosechem., 1927, 8, 16—18).—The average composition of the dry nettle stalk, free from leaves, is cellulose 35%, lignin 23·85%, pentosan 14·13%, alcohol-benzene extract 11·07%, protein 7·21%, ash 6·96%. The lignin obtained by the Willstätter method is very similar to that from pine and other woods. The stalk contains two types of fibre, the bast fibre (20% of the dry stalk), which contains 14% of pentosan but is practically free from lignin, and can be isolated with difficulty by retting, and the lustrous fibre of the inner lignified portion of the stalk, which is removable only by chemical means. The former are 2—6 cm. long; the latter only 0·5—5 mm. The bast fibres cannot readily be separated from the remainder of the stalk by the biochemical methods which give good results with flax, *e.g.*, lactic acid bacteria in dilute alkaline solution. Whilst they are readily isolated by means of sodium hydroxide solution at atmospheric pressure, the incrusted fibres require a higher pressure; and since the bast fibres are attacked during a high-pressure boil a two-stage process is necessary. The sulphite process gives better results, as both fibres are obtained without deterioration, but bleaching is necessary to remove the green colour due to chlorophyll. The dilute nitric acid isolation method (cf. B., 1925, 68) gives a 35—40% yield of fibre containing only 3—4% of pentosan, but the method suffers from the disadvantage that hydrocyanic acid is formed in considerable quantities. The alternative treatment of the stalk with chlorine and 5% sodium hydroxide solution gives without injury to the bast fibres a lignin-free fibre which still contains 14% of pentosan, as the latter is only slowly removed by this method. Further treatment lowers the pentosan content, renders the fibre hard and brittle, and increases the copper number, the final product having a copper number of 4·55 and a pentosan content of 2·8%. On hydrolysis with 72% sulphuric acid the fibre containing 14% of pentosan yields total sugars (calculated as dextrose) 103·5% (theory 111·4%); reducing sugars 81·94% (theory 85·61%). Nettle cellulose is less resistant to sodium hydroxide, carbonate, and sulphide, but is less rapidly attacked by nitric acid than cotton under the same conditions. With acetic anhydride and zinc chloride or sulphuric acid it forms a triacetate in good yield, having acetyl content 41·70—43·28%, $[\alpha]_D^{25}$ in chloroform solution —16·6° to —18·8°, mol. wt. by camphor method 1200—1500. The results agree closely with those given by cotton cellulose triacetate. W. J. Powell.

Electrical method for the determination of moisture in sulphite cellulose. E. Schlumberger (Papier-Fabr., 1927, 25, 81—83).—The change in electrical resistance of wood cellulose with moisture content provides a rapid and simple method for moisture determination. The resistance of a sample, placed between two plate electrodes, is measured in the usual way, the upper plate providing the constant pressure necessary for uniform results. The moisture content of a given pulp, within 1—2%, can thus be readily ascertained. W. J. Powell.

Patents.

Production of washable and fadeless ornamented materials. H. Levy-Diem (E.P. 264,021, 23.1.26).—The material is coated with, or steeped in, a substance sensitive to light, the colours are applied in a thin layer, the material is exposed to light or to the action of any agent which precipitates silver from silver nitrate, and subsequently fixed as in photography with or without development. Alternatively, the colour may be mixed with the light-sensitive substance and applied to the material which is then exposed and fixed. Fabric so prepared may be washed with hot water, calendered, or exposed to sunlight without fading. B. P. Ridge.

Decomposition of bast fibres. G. Frank (G.P. 431,862, 23.7.24).—Before the bleaching proper, the fibres are treated with a cold concentrated solution of a magnesium salt, especially the chloride, the decomposition and bleaching actions being thereby considerably increased. B. P. Ridge.

Retting of flax and similar fibrous plants. I. G. Farbenind. A.-G., Assees. of G. Pfützer and O. Flieg (G.P. 433,366, 24.3.25. Addn. to 411,697; B., 1925, 626).—Carbamide and mineral salts, especially alkali carbonates, are added to the retting water. B. P. Ridge.

Production of artificial silk yarns etc. J. A. Grand (E.P. 253,547, 11.6.26. Conv., 13.6.25).—Threads of artificial silk (particularly from waste material) are cut up and intimately mixed, prior to spinning, with vegetable fibres such as hemp, flax, jute, nettle, etc., previously freed from gum, and mercerised or similarly treated in order to give a silky appearance. B. P. Ridge.

Manufacture of fine organzines and crêpe fabrics in artificial [nitrocellulose] silk. J. Delpech (E.P. 258,571, 4.9.26. Conv., 17.9.25).—Nitrocellulose silk has greater elasticity and tensile strength before denitration than after, hence crêpe fabrics are woven from yarns twisted and doubled before denitration, the process of denitration being carried out on the fabric after crêping. A. J. Hall.

Production of artificial fibres from viscose, immediately washable with water. La Soie d'Aubenton (F.P. 607,266, 11.3.25).—The fibres are precipitated in neutral or weakly alkaline baths to which so much zinc oxide has been added that their zinc content is 2—6%, and are then fixed at 90—120° in a moist atmosphere. L. A. Coles.

Manufacture of filaments and films from viscose. W. Mendel, Assr. to S. A. Neidich (U.S.P. 1,614,431, 11.1.27. Appl., 9.5.25).—Viscose is coagulated in an aqueous solution of sodium phosphate. A semi-solid composition, free from deleterious sulphur compounds and consisting chiefly of cellulose hydrate, is formed by coagulating liquid cellulose thiocarbonate in an aqueous solution containing an acid coagulating agent together with sodium phosphate in such proportions that the said compounds are extracted and held in solution. B. P. Ridge.

Process and apparatus for the production of artificial threads from cellulose derivatives. Ver. Glanzstoff-Fabr. A.-G. (E.P. 249,141, 11.3.26. Conv., 12.3.25).—Filaments from a nozzle projecting into the top of an unheated spinning cell are drawn vertically downwards to the bottom of the cell, a current of air being

meanwhile passed through it in the same direction, thence to a separate closed heating cell in which the last traces of solvent are removed. The filaments are finally reeled or twisted either inside or outside the second cell. B. P. Ridge.

Artificial silk. E. H. Trachsler (Swiss P. 114,677, 22.5.24).—The material consisting of a cellulose derivative (cellulose acetate) in filament form, and still in a swollen condition as a gel, is submitted, under tension, to a cooling treatment and then dried under tension. B. P. Ridge.

Artificial silk. Cuprum (Soc. Anon.) (F.P. 607,965, 14.12.25. Conv., 15.12.24).—The cuprammonium cellulose solution issuing from a spinning nozzle is passed into a bath of water at the ordinary temperature, and the thread so formed subsequently led into a similar bath at 50°. B. P. Ridge.

Manufacture of hollow articles from viscose and similar cellulose solutions. Wolff & Co., E. Czapek, and R. Weingand (G.P. 421,382, 16.10.21).—The interior of a corresponding hollow mould is filled with the initial solution which is precipitated with a precipitating agent, the article is removed in a wet condition, rendered insoluble, washed, and then dried in the inflated state. Thin-walled cellulose articles are easily removed from the mould, and drying over a form is unnecessary. Completely closed articles are produced by leaving only a small opening for the introduction of the precipitating agent and subsequently closing it with a drop of solution. B. P. Ridge.

Apparatus for squirting materials which become plastic on heating. O. Gergacsevics (G.P. 431,537, 12.7.25).—The feeding cylinder is divided by means of an insulating partition into two zones, the upper of which is cooled, and within which a forcing piston works, while the lower zone is heated and slowly melts the cold material forced into it, the plastic material so formed being forced through heated narrow channels and stored in a high-pressure cylinder, which is also placed in the heated zone. From the latter the material is squirted into the form by a piston acting under higher pressure. Continuous working is possible with this arrangement. B. P. Ridge.

Pulp from resinous wood. A. R. Subervie and R. Dulou (F.P. 607,450, 4.12.25).—The wood (maritime pine) in the form of thin slices is swollen by means of steam, sodium carbonate, and slaked lime, and the wood oils are then expelled by steam. B. P. Ridge.

Wood pulp. W. D. Mount (F.P. 607,726, 15.10.25).—Black sodium carbonate and lime are mixed in a liquid and causticised, the product is separated, and wood treated with the clear liquid. B. P. Ridge.

Manufacture of new products comprising lignone derivatives. C. F. Cross and A. Engelstad (E.P. 263,520, 21.9.25; cf. E.P. 216,949; B., 1924, 707).—Concentrated solutions of lignone derivatives obtained in the manufacture of wood pulp are mixed with hydrocarbons of high mol. wt. or with solutions of such hydrocarbons; condensed or oxidised before such admixture; treated with chromic acid and mixed with finely-divided carbon; condensed and caused to dissolve a mineral oxide such as ferric hydroxide; or mixed with hydrocarbons and bitumen, cotton-seed pitch, oils, or fats, thereby yielding materials suitable as insecticides, protective paints, or printing inks. B. P. Ridge.

Manufacture of paper for electric cables. Felten u. Guilleaume Carlswerk A.-G. (Austr. P. 103,613, 16.3.25).—In the manufacture of pergamin paper suitable for use in insulating high-tension cables, more than 50% of the fibre is ground to a viscous mass. The papers contain a high concentration of cellulose and have high dielectric strength. J. S. G. Thomas.

Manufacture of [bead-decorated] threads, yarns, wire, cords, etc. J. C. Vredenburg (E.P. 263,971, 21.10.25).

Method of utilising waste sulphite cellulose lye. C. G. Schwalbe (U.S.P. 1,615,102, 18.1.27. Appl., 21.1.25. Conv., 30.1.24).—See E.P. 228,512; B., 1925, 843.

Dyeing artificial silk (E.P. 263,222).—See VI.

Dyeing cellulose acetate (E.P. 263,579).—See VI.

Pattern effects on cellulose acetate (E.P. 263,248).—See VI.

Printing on cellulose acetate (E.P. 263,355).—See VI.

Basic copper sulphate for cellulose solutions (E.P. 260,212).—See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Control of bleach liquors. Rys.—See VII.

Patents.

Production of pattern effects on acetyl [cellulose acetate] silk fabric. Tootal Broadhurst Lee Co., Ltd., and R. P. Foulds (E.P. 263,248, 9.10.25).—Lustre effects on fabric containing cellulose acetate silk are obtained by printing it with thickened pastes containing organic acids or phenols which dissolve or swell the cellulose acetate silk. Suitable swelling agents include phenol, acetic acid, monochloro- and trichloroacetic acids, butyric acid, and particularly lactic acid. *E.g.*, fabric is printed with a paste prepared by heating a mixture of 50 pts. of lactic acid (B.P.) and 5—10 pts. of feculose, allowed to remain at room temperature for 5 min., then washed, and dried. A. J. Hall.

Printing of fabrics and articles made of or containing cellulose esters or ethers. British Celanese, Ltd. From American Cellulose & Chemical Manuf. Co., Ltd. (E.P. 263,355, 12.3.26).—Ornamental effects are produced on fabrics consisting wholly or partly of cellulose ester or ether artificial silk fibres by printing the fabric with a paste consisting of one or more solvents for the artificial silk, an inert powder such as barium sulphate or calcium carbonate, and a thickening agent such as dextrin, starch, and casein, then maintaining it at 50—125°, and afterwards washing it in water, whereby the printed silk is cleanly removed from the fabric. *E.g.*, fabric consisting of a cotton warp and a weft containing cellulose acetate silk is printed on one or both sides with a paste consisting

of 30 pts. of lactic acid, 3 pts. of water, 5 pts. of dextrin, and 12 pts. of infusorial earth, then maintained at 50—125° until, in the succeeding washing in water, the printed parts become detached. Artificial silk fibres suitable for this process include those prepared from cellulose acetate, propionate, and butyrate, nitrocellulose, ethyl-, methyl-, and benzyl-cellulose. Suitable solvents for use in printing pastes include acetone and acetic and lactic acids (cf. E.P. 263,248; preceding). A. J. Hall.

Dyeing acetylcellulose [cellulose acetate.] O. Y. Imray. From Soc. Chem. Ind. in Basle (E.P. 263,579, 28.10.25).—Suitable water-insoluble dyes for cellulose acetate silk are prepared in a solubilised form (cf. E.P. 219,349; B., 1924, 906) by admixture with sulphite-cellulose waste lyes, and may then be employed for dyeing by the methods usually adopted for such dyes. *E.g.*, a satisfactory dye liquor is prepared by adding 0·5 pt. of a paste containing 20 pts. of *o*-aminoazotoluene and 80 pts. of a 50% sulphite-cellulose lye to a 0·2% soap solution; dyeing is effected at 75°. A. J. Hall.

Dyeing artificial silk [with logwood]. C. S. Bedford (E.P. 263,222, 21.9.25).—Viscose and cellulose acetate silks are dyed in black shades by treatment with an aqueous solution of logwood and then after-chromed, both fibres having a substantive affinity for logwood. Viscose silk may be dyed cold, whereas cellulose acetate silk requires the dye bath to be maintained at 60—80°. *E.g.*, cellulose acetate silk is immersed in a 2—5% solution of logwood at 70—80° for 15—30 min., whereby it acquires a light orange to dark brown shade, and is then developed in a 1—3% solution of sodium dichromate at 60° for 10—30 min. A. J. Hall.

Printing with basic dyestuffs. P. P. Sazanoff (E.P. 262,849, 11.9.25).—Basic dyes may be used together with mordants for them, such as sulphurised phenols (Katanol), direct and sulphur dyes, in printing pastes without premature formation of the insoluble colour lake, provided that the basic dye is present in the form of its free base or leuco-derivative of the base. The further addition of an alkali to the printing paste maintains the basic dye in the form of its base, and the addition of soap increases the purity of the resulting shade. A satisfactory paste for printing white or naphthol-prepared fabrics consists of 10 pts. of Rhodamine B, 200 pts. of water, 685 pts. of 8% solution of gum tragacanth, 30 pts. of a solution of sodium carbonate (*d* 1·36), 50 pts. of a 50% solution of Katanol, and 25 pts. of Turkey-red oil. When Thionine Blue GO is used instead of Rhodamine B a small quantity of sodium hyposulphite is added to maintain the dye base in its leuco-form. A. J. Hall.

Colouring of plastic materials. Badische Anilin- u. Soda-Fabr. (E.P. 242,274, 26.10.25. Conv., 3.11.24).—Plastic materials (cellulose derivatives are excluded) such as waxes, solid paraffin, rubber, resins, etc. are mechanically worked (rolled or kneaded) with colouring substances until the colour particles are so finely dispersed that they are in substantially colloidal solution in the plastic material. At the conclusion of the process no colour particles should settle from a solution of the plastic material in a solvent not capable of dissolving the dye. *E.g.*, a powdered mixture containing 100 pts. of unvulcanised rubber and 15 pts. of Indanthrene Blue GGSL is passed 20—30 times between heated friction rollers, so that the dye does not settle from a solution in benzene of the product even after several months. A. J. Hall.

Washable silk piece goods and process of dyeing it. J. Seyer, Assr. to Standard Silk Dyeing Co. (U.S.P. 1,615,520, 25.1.27. Appl., 24.2.23 and 14.6.26).—See E.P. 207,776 and 219,697; B., 1924, 1010.

Process for the preparation of printing paste. H. Müller, Assr. to J. R. Geigy A.-G. (U.S.P. 1,615,908, 1.2.27. Appl., 22.1.26. Conv., 22.10.23).—See E.P. 223,888; B., 1926, 124.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Catalytic oxidation of ammonia. W. W. Scott and W. D. Leech (Ind. Eng. Chem., 1927, 19, 170—173).—The use of cobalt oxide as catalyst for the oxidation of ammonia gas was studied alone and with promoters. The catalyst was in all cases prepared by evaporating a solution of the nitrates and calcining at 230°. It was used in a granular form, without carrier, packed in a Gooch crucible within a silica conversion tube. The working temperature was 690—800°. The gas-flow speed was adjusted to the optimum for each catalyst. The conversion efficiency for cobalt oxide alone was found to be 79·3%. Amongst numerous promoters tried, the best results were given by bismuth (3%) and aluminium (10—12%). The conversion efficiencies of mixtures of cobalt oxide with different proportions of the oxides of these two metals were studied in detail. With optimum proportions the efficiencies are well over 90%. The capacity of 1 lb. of the catalyst is 60 lb. of anhydrous ammonia per 24 hrs. Extreme purity of the catalyst is unnecessary, nickel and iron, for example, being harmless. All the metal promoters which increased efficiency have m.p. below that of cobalt, whilst those of higher m.p. were injurious. The physical structure of the catalyst has a marked bearing on the results obtained. C. Irwin.

Manufacture of Glauber's salt and of anhydrous sodium sulphate from residues left after the concentration of potassium salts. K. Koelichen (Rocz. Chem., 1926, 6, 711—727).—The systems $MgSO_4$–$NaCl$–H_2O and $MgSO_4$–Na_2SO_4–H_2O are examined with a view of finding the most rational method for the preparation of sodium sulphate by the refrigeration of residues from the Zechstein (Germany) potassium salt deposits. Addition of a small excess of sodium chloride to the solution of residues (sodium chloride and kieserite) prevents supersaturation of sodium sulphate, thereby increasing the yield of the latter. The best results are obtained by extracting astrakhanite with 20% sodium chloride solution. Anhydrous sodium sulphate can best be prepared from the decahydrate by fusing at 70° a mixture of 198 pts. of the latter, 40 pts. of sodium chloride, and 32 pts. of astrakhanite, when 99·3% yields of the anhydrous sulphate may be obtained. R. Truszkowski.

Dehydration of the crystallohydrates of metallic salts. M. A. Rakusin and D. A. Brodski (Z. angew. Chem., 1927, **40**, 110—115; cf. B., 1926, 1011).—The cold dehydration of a number of crystallohydrates was further studied. Placed in a desiccator over sulphuric acid, borax loses 5 mols. of water in 65 days and dehydration continues. Sodium carbonate loses 9 mols. in 5 days, but the last mol. is only partly removed in 65 days. Glauber's salt is completely dehydrated in 4 days. Of other sulphates tested, magnesium sulphate loses 5 mols. of water in 45 days over sulphuric acid, zinc sulphate, 6 mols. in 40 days; nickel sulphate, 1 mol. in 18 days; cobalt sulphate, 5 mols. in 70 days; and copper sulphate is unaltered. These results are compared with those given by dehydration over alcohol, drying in stationary air and in a current of air; while in the case of unstable hydrates the effect of drying in a current of air is more rapid, in most cases dehydration over sulphuric acid proceeds further. The evidence that all the molecules of water are not grouped similarly with regard to the salt is particularly strong with sodium carbonate and nickel sulphate. C. Irwin.

Volumetric determination of alumina in aluminium salts. F. G. Germuth (Ind. Eng. Chem., 1927, **19**, 144—145).—1 g. of the salt is dissolved in 100 c.c. of water, methyl red indicator is added and a slight excess of ammonia solution. The excess ammonia is boiled off as indicated by the colour change, the aluminium hydroxide filtered through a Gooch crucible and washed with ammonium chloride solution. Excess of standard sulphuric acid is added to the filter, the aluminium hydroxide being dissolved, and the excess of acid determined by titration with potassium hydroxide and methyl orange. If iron is present in the sample, it must be separately determined and deducted. The accuracy is equal to that of the gravimetric method. C. Irwin.

Standardisation of titanous sulphate solutions. W. M. Thornton, jun., and A. E. Wood (Ind. Eng. Chem., 1927, **19**, 150—154).—The apparatus used for the storage of titanous sulphate solution under hydrogen at a pressure of 12 in. of water is described. The use of rubber connexions was found to lead to a slight error, possibly owing to penetration by the oxygen of the air in spite of all precautions. Potassium thiocyanate was found reliable as an internal indicator, giving good agreement with results by electrometric titration. Ferric alum, any ferrous iron present being carefully oxidised with $0 \cdot 05N$-potassium permanganate after solution, was found to be a satisfactory secondary standard. Ferrous ammonium sulphate purchased as pure was found to show variation between different samples, and was therefore inadmissible as a primary standard. "Sibley iron ore" from the U.S. Bureau of Standards was preferred for this purpose. The variation in the standard titanous sulphate solution over a period of 3 months as determined by these methods was 0·22%. C. Irwin.

Determination of nitrogen in nitrates. A. Pizzarelli (Staz. sper. agr. ital., 1926, **59**, 139—142; Chem. Zentr., 1926, II, 1773—1774).—20 c.c. of a solution containing 16·5 g. of sodium nitrate or 20·0 g. of potassium nitrate per litre are heated with 10 c.c. of hydrochloric acid, d 1·1, and 20 c.c. of ferrous chloride solution, prepared by dissolving 200 g. of piano wire in hydrochloric acid and diluting to 1 litre. The nitric oxide liberated is washed with 20 c.c. of 10% sodium hydroxide, and collected over water. The percentage of nitrogen $= 100Ka/b$, where a and b are the volumes of nitric oxide obtained from the nitrate under test and from the pure salt respectively, and $K = 0 \cdot 1647$ and $0 \cdot 1387$ for sodium nitrate and potassium nitrate respectively. L. A. Coles.

Control of the manufacture of bleach liquors. L. Rys (Paper Trade J., 1926, **83**, 51—52; Chem. Abstr., 1926, **20**, 3566).—An adaptation of Votocek's method for chlorides (A., 1918, ii, 238, 272) to the determination of chlorates. The sample is diluted to 100 c.c., 5 c.c. of 10% sodium nitrite solution and 10 c.c. of concentrated nitric acid are added. After 1 hr., 1 c.c. of hydrogen peroxide is added. After 15 min. the solution is diluted to 175 c.c., titrated with $0 \cdot 1N$-mercuric chloride in presence of 6 drops of a solution of sodium nitroprusside (6 g.) and nitric acid (10 drops) in water (30 c.c.), giving the total chlorine as chloride. Hypochlorite is separately determined by oxidising the sample, diluted to 175 c.c., with hydrogen peroxide, acidifying with nitric acid, and titrating with mercuric chloride. A. A. Eldridge.

Concentration of Canadian flake graphite ores. C.S. Parsons (Canad. Dept. Mines, 1925, [670], 109—118).—Canadian graphite ores contain 7—15% of graphite, and in concentrating them the basic principle is to obtain the maximum recovery of coarse flake (+ 60-mesh) in a high-grade concentrate suitable for treating in the ordinary refining plant. For grinding the ore a cylindrical rod mill operating on a 1 : 1 pulp ratio gives the best results; the overflow from the mill passes to a Callow "rougher" cell the concentrate from which passes to a "cleaner" cell, both cells being operated with kerosene or steam-distilled pine oil. The concentrate is treated on tables for the removal of entangled sand and sand particles containing fine graphite. The table concentrate is cleaned once more by flotation and table treatment, whilst the tailings from the cleaning processes are reground in rod mills and re-treated as before. After de-watering and drying, the combined concentrates are refined by dry grinding in a burrstone mill followed by screening. A. R. Powell.

Plasticity of finishing limes. Briscoe and Mathers.—See IX.

Patents.

Continuous apparatus for the manufacture of caustic soda etc. by causticising. W. D. Mount (F.P. 608,190, 15.9.25).—The apparatus consists of a column formed of superimposed cylinders, which can be evacuated, some of the lower cylinders being separated from one another by plates with a central opening, thereby forming so many disc-shaped reaction spaces. Sodium carbonate is introduced at the top of the column and flows through these reaction spaces through which milk of lime is passed. Hot air or gases are passed through the column from below. B. W. Clarke.

Manufacture of alkali monoxide. Roessler & Hasslacher Chemical Co. (E.P. 253,520, 7.6.26. Conv., 8.6.25).—Alkali metal is added to a solid diluting sub-

stance such as a monoxide of the alkali metal from a previous experiment, or sodium chloride, contained in a rotary kiln, and is oxidised at 120—200° with air, which is so regulated that only a limited amount is in contact at any time with the material. A high degree of dilution is maintained by adding the alkali metal gradually, and before the quantities previously introduced have been fully oxidised. W. G. Carey.

Preparation of a sodium-sulphur compound. F. A. Frazier, Assr. to F. A. Frazier Co. (U.S.P. 1,614,063, 11.1.27. Appl., 23.8.23).—A sodium-sulphur compound containing not less than 60% of sodium polysulphide, and not more than 10% of sodium thiosulphate, together with free sulphur and sodium sulphate, is made by heating together sodium sulphide, sodium carbonate, and sulphur for 1—2 hrs. at 230—280°. W. G. Carey.

Production of ammonium sulphate. Elektrizitätswerk Lonza [Gampel & Basel] (Swiss P. 114,699, 25.11.24).—In the production of ammonium sulphate by the interaction of gypsum, ammonia, and carbon dioxide, 70% or more of the ammonium sulphate formed is precipitated by saturating the solution with ammonia, and removed. Before the mother-liquor is used again in the process excess ammonia is expelled, sufficient being left for the formation of concentrated ammonium sulphate solution. L. A. Coles.

Production of a basic copper sulphate suitable for the manufacture of ammoniacal copper oxide cellulose solutions for the spinning of artificial silk by the stretch-spinning process. J. P. Bemberg A.-G. (E.P. 260,212, 25.6.26. Conv., 20.10.25).—Copper hydroxide or " cement " copper is dissolved in a solution of ammonium sulphate and hydroxide, and the basic sulphate precipitated with sulphuric acid, the ammonium sulphate simultaneously formed being used again for dissolving more initial material. B. P. Ridge.

Production of aluminium sulphate and alumina from clay. M. Lavoye (F.P. 607,537, 8.12.25).—Clay is heated at about 600° in a revolving furnace. The superheated steam generated by evaporation of the water in the clay decomposes the silicates, yielding alumina readily soluble in sulphuric acid. L. A. Coles.

Sodium magnesium carbonate. H. Rubinstein, and Solt & Kronstein (Austr. P. 103,630, 6.11.24).—Magnesium carbonate is treated at 70° for several hours with sodium carbonate solution saturated with sodium bicarbonate, or, alternatively, carbon dioxide is passed continuously into the solution under pressure. L. A. Coles.

Production of pure cerium compounds. Deutsche Gasglühlicht-Auer-Ges.m.b.H. (G.P. 433,454, 19.1.24).—Basic cerium compounds, prepared as a step in the purification of crude cerium compounds, are washed with solutions containing sodium chloride or other salts, or, if necessary, the basic salts are precipitated in such solutions. Didymium and lanthanum compounds can be prepared from the mother-liquor and washings. L. A. Coles.

Manufacture of titanium compounds [sulphate]. P. Spence & Sons, Ltd., A. Kirkham, and H. Spence (E.P. 263,886, 1.7.25).—Crystalline titanyl sulphate tends to yield on drying a tough, fibrous material, but a dense, granular, monohydrated salt is prepared by heating, with agitation, a solution of titanium sulphate having *d* 1·5—1·75 (tested cold), containing sulphuric acid to the extent of 4—12 mol. of SO_3 to each mol. of TiO_2, and maintaining a temperature of 90—130° during crystallisation. The crystallisation is hastened by the addition of previously prepared monohydrated titanyl sulphate. The crystals are washed with dilute sulphuric acid, or a solution of a sulphate such as nitre cake, aluminium sulphate, or titanium sulphate. W. G. Carey.

Manufacture of carbon dioxide. P. E. Haynes (E.P. 263,922, 3.10.25).—A mixture of carbonaceous fuel and a solid carbonate is fed into a rotating reaction vessel heated externally, and the carbon is burnt to carbon dioxide by oxygen, or air enriched with oxygen, the temperature of reaction being kept under control owing to the absorption of heat by the decomposition of the solid carbonate with liberation of additional carbon dioxide. The materials are in heat-exchange relation, the oxygen being heated by passing through thermally decomposed carbonate which is discharged from the bottom of the apparatus, while the carbon dioxide evolved from the reactions heats the oncoming mixture of carbonate and fuel and is thereby cooled. The carbon dioxide is sufficiently pure to be liquefied by moderate cooling and a pressure of about 1000 lb./sq. in. W. G. Carey.

Production of carbon disulphide. W. Buchner (G.P. 433,525, 4.11.25).—In the thermo-electric production of carbon disulphide from its elements, the carbon is stacked between electrodes lowered into the material through two or more shafts connected together by channels and bellied around the ends of the electrodes. Alternatively, the electrodes may be introduced through lateral shafts. The carbon disulphide is withdrawn through an opening below the ends of the electrodes. L. A. Coles.

Sulphur purification. H. H. Wilkinson, Assr. to Union Sulphur Co. (U.S.P. 1,613,632, 11.1.27. Appl., 26.11.23).—Sulphur is heated at below its b.p., a current of inert gas is passed through the molten sulphur, and the gas and sulphur carried thereby are led into a condensing chamber. W. G. Carey.

Desulphurising chemical solutions and precipitates. K. Sagstetter and C. B. Heiler (G.P. 433,522, 14.11.25).—Sulphur and undesirable sulphur compounds are removed from aqueous solutions or suspensions by acidification and the addition of reducing agents, such as iron, tin, zinc, or stannous chloride, followed by heating to expel hydrogen sulphide, provided the treatment has no action upon the desired product. For example, a suspension of freshly-precipitated *blanc fixe* containing sodium sulphides, sodium thiosulphate, and iron sulphide as impurities, prepared by treating barium sulphide with sodium sulphate solution, can be purified by the above process. L. A. Coles.

Desulphurising gases. Production of sulphur from metal sulphides. E. A. Prudhomme (F.P. 607,259—61, 17.2.25).—(A) The gases are passed over heated metals, such as nickel. (B) Sulphur dioxide

and hydrogen sulphide, produced by heating separate portions of the sulphide in air and in reducing gases respectively, are passed simultaneously into metal salt solutions. (c) The sulphide is made the cathode in an electrolytic cell, using dilute sulphuric acid as electrolyte.
L. A. COLES.

Production of bromine and magnesium chloride tetrahydrate from bishopite. PREUSSISCHE BERGWERKS- & HÜTTEN-A.-G. ABTG. SALZ & BRAUNKOHLENWERKE BERLIN, BERGINSPEKTION VIENENBURG, Assees. of P. C. BÜCHNER (G.P. 433,586, 15.10.25).—Bishopite is fused under conditions suitable for hydrolysis of the bromides present and oxidation of the hydrogen bromide formed, the bromine being expelled with the steam, and the residue is heated until it has the constitution, $MgCl_2,4H_2O$. About 3—5% of water or bishopite solution is added to the fused material during the process.
L. A. COLES.

Synthesis of ammonia by hyper-pressures. G. CLAUDE, Assr. to LAZOTE INC. (U.S.P. 1,615,535, 25.1.27. Appl., 19.11.21. Conv., 20.11.20).—See E.P. 171,970; B., 1923, 95 A.

Manufacture of sodium fluoride from hydrofluosilicic acid. W. SIEGEL (E.P. 263,623, 1.1.26).—See U.S.P. 1,581,819; B., 1926, 538.

Process for making barium carbonate. J. E. MARWEDEL and J. LOOSER, Assrs. to RHENANIA VER. CHEM. FABRIKEN A.-G. (U.S.P. 1,615,515, 25.1.27. Appl., 21.10.22. Conv., 4.1.22).—See G.P. 427,223; B., 1926, 630.

Manufacture of carbon disulphide. P. SIEDLER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,615,659, 25.1.27. Appl., 28.11.25).—See E.P. 236,577; B., 1925, 759.

VIII.—GLASS; CERAMICS.

Valency of arsenic and antimony in glass. H. HEINRICHS and F. SALAQUARDA (Glastechn. Ber., 1926, **4**, 130—137; Chem. Zentr., 1926, II, 2008).—The amount of arsenic pentoxide and antimony pentoxide is determined by heating the powdered glass with potassium iodide and hydrochloric acid and determining the excess of iodine. The total arsenic and antimony, including tervalent arsenic, is also determined. From 75 to 80% of the arsenic is present as As_2O_5, but only 0—6% of the antimony as Sb_2O_5. The determination is facilitated by the addition of a large excess of saltpetre.
B. W. CLARKE.

Determination of boric acid in silicates. R. SCHMIDT (Sprechsaal, 1926, **59**, 541—543; Chem. Zentr., 1926, II, 2010).—Hydrochloric acid is added to an alkaline borate solution after the complete removal of the silica, in the presence of methyl orange as an indicator, until the solution is red; the boric acid can then be determined in the usual way, after the addition of glycerin, by titration with caustic soda with phenolphthalein as an indicator. B. W. CLARKE.

PATENTS.

Low-expansion glass. SOC. LA RADIOTECHNIQUE (E.P. 258,872, 21.9.26. Conv., 22.9.25).—Glass fusible with difficulty and having a low coefficient of expansion contains a high proportion, but preferably less than 70%, of silica, the remainder being a mixture of boric and sodium oxides in the ratio of 85 : 15.
B. W. CLARKE.

Glazing ceramic bodies. ORQUALIN-GES.M.B.H. NÜRNBERG, SITZ JENA (G.P. 432,724, 5.12.23).—A liquid energising medium is sprayed or otherwise applied to the green body before firing, so that the surface of the body contains more liquid than the interior, the liquid medium consisting of completely soluble salts, while the batch contains only partly soluble salts.
B. W. CLARKE.

Porcelain crucibles. STAATLICHE PORZELLAN-MANUFAKTUR, and A. KÖNIG (G.P. 433,376, 19.2.25. Addn. to 403,405).—Filtration with the crucibles described in the chief patent (cf. E.P. 220,618; B., 1925, 244) is expedited by the provision of a flange on the porous bottom, constructed of the same material.
L. A. COLES.

Manufacture of unburned refractory brick. A. MARKS, Assr. to U.S. REFINING CO. (U.S.P. 1,616,192, 1.2.27. Appl., 6.5.26).—A magnesitic body material is mixed with a suitable siccative oil, moulded, and dried until hard. B. W. CLARKE.

Production of a sodium-aluminium borosilicate glass. SIBOR S. A., VERRERIES DE ROMONT (E.P. 245,131, 21.12.25. Conv., 21.12.24).—See Swiss P. 114,242; B., 1926, 747.

Removal of glass from glass furnaces. H. SEVERIN (E.P. 240,471, 23.9.25. Conv., 23.9.24).

IX.—BUILDING MATERIALS.

Impregnating wood with paraffin. L. W. EBERLEIN and A. M. BURGESS (Ind. Eng. Chem., 1927, **19**, 87—88).—Impregnation with paraffin greatly increases the resistance of wood to dilute acids and alkalis. The wood is soaked for 12 hrs. in water at room temperature and placed in a tank of paraffin at 71°. The temperature is raised to 105° in 30 min., and then to 135° in 3—4 hrs. When all effervescence has ceased the wood is transferred to another tank at 71°, where it remains for 30 min. It is removed and wiped clean. Up to 124% by wt. of paraffin is absorbed. Such treated wood absorbs as little as 2·8% of moisture after 48 hrs. immersion in 5% sulphuric acid. C. IRWIN.

Studies in wood preservation. C. G. SCHWALBE (Z. angew. Chem., 1927, **40**, 105—110).—An apparatus was devised to study the impregnation of wood on a moderately large scale. Sections of entire stems, with the bark, 1—2 m. long were cut and supported in a vertical position on porcelain rings in stone jars. The upper end was cut away internally to some little depth, leaving an outer ring 1 cm. thick standing up. The liquid to be tested was fed in here from a Mariotte's bottle. It was found that water or dilute ammonia extracted a colloidal reducing material. Wide differences in the time necessary to impregnate undried wood in this way were noticed between different specimens; in general, wood cut in May is more slowly impregnated than winter-felled wood. All the preservatives tested impregnated only the outer part of the undried stems. Suitable chemical tests were devised for showing up the preserva-

tive in a cross-section, *e.g.*, ammonium sulphide for mercuric chloride, hydrogen sulphide for arsenic, potassium thiocyanate and ferric sulphate for sodium fluoride (the colour is discharged), and stannous chloride for dinitrophenol. C. Irwin.

Recent measurements and observations on automatic shaft kilns for calcining cement. H. Richarz (Zement, **15**, 492—496, 507—512, 531—534, 549—552; Chem. Zentr., 1926, II, 2010).—Data are given for the Mannstädt kiln, in which a reducing atmosphere is avoided, including measurements of temperature, draught pressures and quantities, waste-gas temperatures, thermal economy, etc. The shaft kiln can operate with coke residues high in ash, whereas the rotary kiln needs good coal low in ash.

B. W. Clarke.

Plasticity of finishing limes. H. T. Briscoe and F. C. Mathers (Ind. Eng. Chem., 1927, **19**, 88—93).—All quicklimes yield a fairly plastic putty when slaked with excess of water, but very few dry hydrated limes yield a plastic lime putty on addition of water. By "plasticity" is here meant the quality of being readily worked with a plasterer's trowel. For this quality the presence of magnesia is essential. If a plastic putty is dried without dehydration and resoaked, the plasticity suffers. Drying at 325°, at which temperature magnesia is dehydrated, however, increases the plasticity. Steam-treated hydrated lime has unsatisfactory plasticity, and so has lime slaked with excess of water. It is believed that in a plastic hydrated lime the magnesia is not fully hydrated, and the completion of hydration during the formation of the putty produces the desired quality. If limestone is treated with sodium chlordie before burning the rate of hydration is reduced and the plasticity improved. A higher proportion of sodium chloride was found in samples of dolomite known to give plastic limes. A moderate temperature of burning, 1050—1150°, gives the best results. A study of the loss in weight after drying at 325° in various samples tended to confirm the view of plasticity here expressed.

C. Irwin.

Patents.

Making mortars and cements. O. Lindemann (E.P. 252,210, 18.5.26. Conv., 18.5.25).—Finely-ground glass, *e.g.*, glass-furnace waste, bottle glass, etc., is mixed with cement, either during the grinding of the clinker or during the preparation of the mortar, producing increased tensile strength in the set cement.

B. W. Clarke.

Rendering cement receptacles, pipes, walls, blocks, and the like impervious to chemical reagents. J. A. H. Itier (E.P. 247,976, 18.2.26. Conv., 18.2.25).—The cement surface is moistened with a concentrated potassium silicate solution, allowed to dry in the air, and treated with sulphuric acid which dehydrates and heats the silicate coating. The porous layer resulting is soaked for several days in dilute sulphuric acid which deposits calcium sulphate in the pores, and the surface is then treated successively with a solution of sodium pectinate or alginate and a resin soap solution, forming a protective layer resistant to all known reagents.

B. W. Clarke.

Calcining cement, magnesite, lime, and the like. H. Stehmann (G.P. 431,987, 13.7.23. Addn. to 378,364).—A small opening is left between the housing surrounding the rotary furnace, described in the main patent, and the fixed head of the furnace, through which air is drawn; a desirable cooling of the furnace is produced.

B. W. Clarke.

Manufacture of mortar. A.-G. für Steinind., and W. Braun (G.P. 428,431, 15.2.25).—Freshly-burnt, still hot lime is mixed and preferably ground with moist trass, slag, or ashes under steam pressure, and the material heated to expel the water of hydration. The effect of the steam treatment is to increase the binding power of the mixture. B. W. Clarke.

Treatment of mortar-forming materials and mortar. W. Kohen (G.P. 432,723, 9.8.23).—Ferric oxide in larger quantities than usual is mixed with cement, with the addition of sulphates, *e.g.*, calcium sulphate, and aluminous compounds, the materials to be mixed with the cement being exposed to damp air for some time before mixing. B. W. Clarke.

Artificial stones. B. Hessellwitz and M. Contin-souza (F.P. 606,952, 9.3.25).—A 60% aqueous solution of magnesium sulphate is saturated with crystalline magnesium chloride, and a mixture of 1 pt. of magnesium oxide and 4 pts. of calcium carbonate added until a pasty mass is formed which can still just be poured into moulds, where it is allowed to set. Other magnesium salts can also be employed. B. W. Clarke.

Impregnating wood with colour stains fast to light. G. Schmalzl (Austr. P. 102,663, 13.5.24).—Wood impregnated with staining solution is subjected to the action of the Röntgen rays. For example, maple or pine logs are stained blue by successive impregnation with potassium ferrocyanide solution, air-drying, 10—40 min. exposure to the rays, washing with water, and fixing with silver nitrate solution. Grained or other effects are produced by covering the wood with patterns cut from sheet lead or other material impervious to the rays, while the wood is exposed to their action.

L. A. Coles.

Fireproofing composition. O. Petereit (G.P. 433,478, 1.2.25).—The composition, consisting of a suspension of colloidal alumina and silicic acid, together with filling material, in a solution containing alkali sulphite, is prepared by passing ammonia into a solution containing aluminium sulphate and aluminium sulphite to precipitate alumina, and then adding water-glass and filling material, such as barium carbonate, ground quartz, fluorspar, or lithopone. L. A. Coles.

Impregnation of unpaved roads. "Allchemin" Allgem. Chem. Ind. A.-G., and R. Lichtenstern (E.P. 263,975, 3.11.25).—A colloidal solution of naphthene soaps in distillation residues rich in paraffin is applied to the road surface, forming an impervious layer of lime soap by reaction with the constituents of the road, having an oily surface which acts as a dust-binding medium. B. W. Clarke.

Manufacture of fused cement and apparatus therefor. G. Dumas, Assr. to Soc. Anon. des Chaux et Ciments de Lafarge et du Teil (U.S.P. 1,615,260,

25.1.27. Appl., 15.1.24. Conv., 29.9.23).—See E.P. 222,426; B., 1924, 1014.

Bituminous emulsion (F.P. 608,123).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

The equilibrium $C + CO_2 \rightleftharpoons 2CO$. Thermodynamics of the blast-furnace process. V. Falcke (Z. Elektrochem., 1927, 33, 1—11).—Values recorded in the literature for the equilibrium constant, $k = [CO]^2/[CO_2]$, at various temperatures (523—1150°) have been collected and compared by plotting on a log k–T diagram: corresponding values for the heat of reaction, q, have been calculated from those of k. Independent sets of values for k and q have been calculated from sp. heat data and figures for the heats of combustion of carbon and carbon monoxide. At a given temperature two values of k (calculated), and similarly of q (calculated), are obtained according as graphite or amorphous carbon is regarded as participating in the reaction; but for k (calculated) the difference becomes inappreciable at the higher temperatures. Above 750° values of k (experimental) agree well among themselves and with the corresponding calculated values, but at lower temperatures they are somewhat irregular. Comparison of values of q (from experimental k) with those of q (calculated) shows that above about 750° the former fit in with the assumption that graphite participates in the equilibrium, whereas at lower temperatures they correspond more closely to values for amorphous carbon. Values of k (experimental) obtained from equilibria reached from the carbon monoxide side in the presence of iron powder diverge notably from those obtained in other ways, when the equilibrium temperature is below 750°. This discrepancy is ascribed to the abnormal condition of the carbon in the iron; it is markedly reduced if the iron is previously treated with carbon monoxide for a long time, so that free carbon may be deposited in it. In any case, the establishment of equilibrium is undoubtedly slow at the lower temperatures, and is not well defined. Measurements of k derived from the composition of blast-furnace gases have also been collected on log k–T diagrams. They are very irregular at lower temperatures, but correspond approximately with the experimental and calculated values at temperatures above about 750°. Thus the equilibrium $C + CO_2 \rightleftharpoons 2CO$ is only attained in the blast-furnace gases at these higher temperatures.

H. J. T. Ellingham.

Effect of phosphorus on the resistance of low-carbon steel to repeated alternating stresses. F. F. McIntosh and W. L. Cockrell (Carnegie Inst. Tech., Min. Met. Invest. Bull., 1925, 25, 1—28; Chem. Abstr., 1926, 20, 3433).—The addition of phosphorus (0·010—0·125%) to open-hearth steel containing less than 0·15% C increases the endurance of the metal against repeated alternating stresses, increases its hardness, ultimate strength, and elastic limit, has no bad effect on the resistance to shock or vibratory strain, and increases the resistance to corrosion and abrasion.

A. A. Eldridge.

Nickel affects grey iron. T. H. Wickenden and J. S. Vanick (Foundry, 1926, 54, 689—690; Chem. Abstr., 1926, 20, 3420).—Nickel above 1% reduces the combined carbon to 0·8% in cast-iron, and above 5% reduces the total carbon. Nickel makes the pearlite more sorbitic, and prevents the formation of a cementite network by chromium. With carbon above 0·5% the strength is increased by nickel alone. Nickel improves deflexion and toughness; shrinkage and fluidity are not affected. 5—12% Ni confers martensitic hardness; with more than 15% the iron is austenitic.

A. A. Eldridge.

Analysis of steels. W. Singleton (Chem. Age, 1927, 16, Met. Sect., 9—11).—To obviate prolonged boiling to destroy the excess of permanganate used in the oxidation of chromium to chromic acid in the analysis of chromium steels, manganese sulphate is added, followed by ammonia, whereby the permanganate is immediately reduced to manganese dioxide. After acidifying with sulphuric acid, an aliquot part of the solution is filtered through a dry paper and the chromate determined as usual. In low-chromium steels oxidation may be effected by using only a slight excess of permanganate if the solution contains 4 g. of ammonium phosphate in 150 c.c.; the excess permanganate is then destroyed with 5 c.c. of hydrochloric acid. For the colorimetric determination of manganese in steel by the persulphate method, comparison of test and standard is simplified by filtering the light through a nickel sulphate solution. For the determination of molybdenum, 3 g. of steel are dissolved in 200 c.c. of 3% sulphuric acid with the addition of ammonium persulphate until the liquid begins to turn brown; the solution is saturated with hydrogen sulphide and the washed precipitate heated, together with the filter paper, until the latter is completely charred. The residue is fused in a covered crucible with 2 g. of sodium carbonate and the melt leached; the solution is filtered and the filtrate acidified and reduced in a Jones reductor, as usual. For the determination of small amounts of copper in steel, the sulphide, precipitated from a nearly neutral solution, is fused with potassium hydrogen sulphate and the copper determined by the iodide method in the solution of the melt, after addition of 20 c.c. of 10% potassium fluoride solution.

A. R. Powell.

Determination of carbon in cast iron. J. T. Mackenzie (Iron Age, 1926, 118, 415—416; Chem. Zentr., 1926, II, 1994).—In a simplified apparatus for carrying out the determination by the combustion method, suitable for use in works' laboratories, the vessel containing the chromic-sulphuric acid mixture is so constructed that it cannot overflow, and can be filled and emptied without detachment. A Fleming flask containing ascarite in the lower and phosphorus pentoxide in the upper portion is attached to the apparatus.

L. A. Coles.

Determination of manganese in rich alloys. E. de Luisi (Met. italiana, 1925, 17, 464—468; Chem. Abstr., 1926, 20, 3407).—The gravimetric method, which is preferred as a standard, is rapid if the iron is separated in the cold with cupferron. The Volhard–Wolff method should be carried out at 40° with energetic stirring. The bismuthate method is exact; the iron alloy may be decomposed with sodium peroxide. A. A. Eldridge.

Rapid determination of silicon in 8—17% ferro-silicons. G. T. DOUGHERTY (Ind. Eng. Chem., 1927, **19**, 165—167).—The alloys may be decomposed without fusion by a mixture of 10% of nitric acid and 90% of hydrochloric acid. 100 c.c. of the mixed acids and 1 g. of the sample are boiled for ½—1 hr., diluted with cold water, stirred, the precipitate filtered at once, and washed alternately with cold water and hot hydrochloric acid solution (1 : 3). The residue of silica is ignited at a low temperature until nearly white and then at a higher temperature. It is cooled and weighed, and 0·25% is added to the percentage of silicon found in the sample to allow for the silicon usually remaining dissolved in the filtrates. This method is rapid, taking approximately 3 hrs. as against 2 days for the standard fusion method. No silicon carbide was found in the non-volatile residue from a 17·22% sample after ignition with hydrofluoric and sulphuric acids.

M. E. NOTTAGE.

Effect of velocity on corrosion of steel under water. R. P. RUSSELL, E. L. CHAPPELL, and A. WHITE (Ind. Eng. Chem., 1927, **19**, 65—68).—Test pieces of a low-carbon steel, having polished, rough, and rusted surfaces respectively, were suspended in tubes through which tap-water was passed, the diameters of the tubes being such as to give a velocity range covered by that used by earlier workers. The corrosion rates were determined by weighing the test-pieces before and after each run, the duration of the runs varying from 48 hrs. to 2 weeks. Owing to the large volume of water passed through the tubes and the relatively small area of metal surface exposed, the oxygen content of the water remained practically constant. It was found that with uncorroded steel surfaces the corrosion at first increases with velocity up to a maximum and then decreases. The corrosion of highly polished steel is less at very high than at very low velocities. The effect of roughening the surface is to shift the point of maximum corrosion to a point of higher water velocity, and also to cause the corrosion rates observed at high velocities to be greater than those at very low velocities. In all tests rust appeared on the metal surfaces. Similar results were obtained by rotating vertical cylindrical test-pieces at surface speeds up to 8 ft./sec. in distilled water. The corrosion was lowest when the surface speed was practically zero. Over a wide range the effect of increasing the velocity (up to 4 ft./sec.) is to increase the rate of corrosion of rusted steels. Thus, by suitably altering the surface condition of a steel, it is possible to reproduce the apparently conflicting data of earlier workers (cf. B., 1923, 272 A; 1925, 505, 506).

M. E. NOTTAGE.

Oxidation of iron by steam, air, and carbon dioxide at high temperatures. P. P. FEDOTÉEV and T. W. PETRENKO (J. Russ. Phys. Chem. Soc., 1926, **58**, 222—228).—The action of steam, dry air, and carbon dioxide at 1000—1100° on iron rods was investigated with a view to the preparation of so-called "insoluble anodes." The rods, after definite periods of oxidation, were analysed, and sections, etched with hydrochloric acid and polished, were examined micrographically. At first large crystals are found which, after 24 hrs.' oxidation, disintegrate into microscopic ones. Only single-phase systems seem to be present, *i.e.*, isomorphous mixtures and solid solution are formed. The mechanism of the oxidation is probably $Fe \rightarrow FeO \rightarrow Fe_3O_4$, the last two forming a solid solution. The final product is Fe_3O_4. With air, Fe_2O_3 is the final product, the iron oxide being very friable. The oxidation, as far as Fe_3O_4, proceeds as with steam, when surface Fe_3O_4 is oxidised to Fe_2O_3, forming a porous layer, which allows more Fe_3O_4 to react, and so on. Mixtures of air and steam oxidise the iron in the stages $FeO \rightarrow Fe_3O_4 \rightarrow Fe_2O_3$, the last two oxides forming solid solutions, which are the final products, depending in composition on the partial pressure of the oxygen. Carbon dioxide, in general, has the same effect as steam.

M. ZVEGINTZOV.

Concentration of Canadian molybdenite ore. W. B. TIMM and C. S. PARSONS (Canad. Dept. Mines, 1925, [670], 96—108).—Molybdenite ores should be ground to pass 35 mesh before flotation. Good recoveries may be effected in Callow cells using pine oil for frothing and kerosene as the collector, a 1% ore requiring about 0·25 lb./ton of the former and 0·5—0·75 lb. of the latter. A pulp ratio of 1 : 1 gives the best results provided that ample "rougher" capacity is provided to make a clean tailing. On the other hand, the capacity of the "cleaner" cells should be kept at a minimum so as to obtaiu a high-grade concentrate by treatment in two successive cells. Pyrites and other floatable impurities may be eliminated from the concentrates by screening on an 80-mesh screen. Examples of the results to be expected from treating four different types of ore are given.

A. R. POWELL.

Trend of flotation. A. J. WEINIG and I. A. PALMER (Quart. Colorado School Mines, 1926, **21**, [2], 5—62).—A description is given of the development of flotation processes and of modern technique as practised at many American treatment plants. The function of the various reagents used in flotation is briefly discussed, and data are given of the annual consumption of these reagents in the United States, of the tonnages of ore milled and of concentrates produced, of working costs for some typical ores, of the royalties demanded by the patentees of the most important processes, and of the payments and deductions made by the smelters for flotation concentrates.

A. R. POWELL.

Finishing process for aluminium and its alloys. L. GUILLET (Compt. rend., 1927, **184**, 134—136).—Pacz has shown that if aluminium or an alloy rich in aluminium is tempered in a solution of sodium fluosilicate (5 g.), nickel sulphate (10 g.), and potassium nitrate (25 g.) in 4 litres of water at 70—80°, coloured lines appear which blacken quickly in the bath. The design produced may be varied according to the relative motion with respect to the liquid accorded to the metal. The deposits appear to start from impure spots on the surface, and their extent is strictly limited by electrolytic protection. They are not very tenacious, and are decreased by cold-beating. Corrosion readily starts along the lines of the pattern, with the formation of alumina. The deposits are probably finely-divided nickel, partly oxidised in the bath, since if they are heated for 2 hrs. at 450° in a current of pure hydrogen they become dark in colour and lose weight to a greater

extent than an untreated sample, owing to reduction to nickel. J. Grant.

Effect of oxygen concentration on the corrosion of copper by non-oxidising acids. R. P. Russell and A. White (Ind. Eng. Chem., 1927, 19, 116—118).—Two samples of copper were used, one containing copper oxide, the other practically oxygen-free. These were immersed in $1 \cdot 2N$-solutions of sulphuric, acetic, and hydrochloric acids, the concentration of dissolved oxygen being varied by saturating the acid solutions with atmospheres containing 0—25% O. All tests were made at 23°. It was found that the rates of corrosion were directly proportional to the concentration of dissolved oxygen in the three acids. For a given oxygen concentration, the rates were the same in sulphuric and acetic acids, but much higher in hydrochloric acid, probably owing to the presence of an additional oxidising agent, viz., cupric chloride. The rates of corrosion in oxygen-free acid were practically zero for oxide-free copper, but the copper containing oxygen always showed some corrosion, probably due to the solution of some of the exposed oxide particles. M. E. Nottage.

Rubber as a solution of corrosion and abrasion problems. H. E. Fritz (Ind. Eng. Chem., 1927, 19, 130—132).—The Vulcalock process for attaching rubber to metals and other rigid constructional materials, in which the pliable, semi-plastic gum is applied to the cleaned material and cured in place, whereby a remarkable adhesion is set up, gives a new chemical engineering material which incorporates the strength and workability of metal with the abrasion- and corrosion-resisting properties of rubber. The material is especially useful for the manufacture of tanks etc. for the transport of corrosive acids. The application of the Vulcalock process is not generally recommended for use where the temperature exceeds 82°. M. E. Nottage.

Still for the purification of mercury. H. S. Booth and N. C. Jones (Ind. Eng. Chem., 1927, 19, 104—107).—The detailed construction, based on accepted principles, of an unbreakable mercury still, made of glass-enamelled iron or steel, is described. It is capable of distilling 3—5 kg./hr. of mercury, and runs for 3—4 hrs. without attention. All parts are arranged concentrically for space economy. The mercury is distilled *in vacuo*, heat being applied either by means of a gas-ring or electrically. The apparatus is provided with an arrangement for bubbling air through the boiling mercury to oxidise base metals. The mercury is subjected to a preliminary purification by filtration and agitation with a solution of mercurous nitrate, followed by washing and drying at 110—120°, before being distilled. M. E. Nottage.

Test for platinum. F. Durdik (Chem. Listy, 1927, 21, 10—12).—The author discusses the touchstone test for platinum and suggests the following modification. Before use, the stone is heated at 110—120° in an air bath, placed in a wooden support, and coated with almond oil. Streaks are then made on the hot stone with the sample to be tested and with a standard, and the behaviour of these streaks towards aqua regia is examined. A. R. Powell.

Metallic thorium. J. W. Marden and H. C. Rentschler (Ind. Eng. Chem., 1927, 19, 97—103).—The history of the preparation of thorium powder and methods used by earlier workers for sintering, fusing, and working the metal are reviewed and discussed. The methods used by the authors, viz., reduction of the chloride with either sodium or calcium, and reduction of the oxide with calcium chloride and either sodium or calcium, showed the last of these methods to be the best, a 90% yield of metal containing 98—100% Th being obtained; full details of the process are given. In the method finally adopted for working the metal the thorium powder was poured into a copper tube and rolled down to a small diameter. The copper-coated thorium thread thus formed was brazed on to tungsten leads and sealed into a flask tubulated at both ends; the copper was then removed by nitric acid, the flask evacuated, and a current passed through the ribbon. In the earlier experiments the thorium powder was pressed into buttons, and the heat-treatment was done in the high-frequency induction furnace, the buttons of thorium being supported on crucibles of pure thoria. The degasification of the metal was accomplished below a red heat. The thorium was then melted or was made workable by heating it just below the fusion point for a few minutes. A method of heat-treatment for the purpose of making wires and slugs for inserts in X-ray targets is described, and various physical properties and constants of the metal are given, together with methods for its analysis. M. E. Nottage.

Determination of thoria in tungsten filaments. D. H. Brophy and C. Van Brunt (Ind. Eng. Chem., 1927, 19, 107—109).—The sample is first burned to oxide by heating it in a quartz tube at 700° in a current of oxygen in an electric furnace. When completely oxidised, an equal proportion of dry gaseous hydrogen chloride is mixed with the oxygen and the tungsten trioxide completely volatilised as oxychloride, the operation taking about 2 hrs. Chloroform vapour may be substituted for the hydrogen chloride with equally good results. The residue is usually regarded as thoria; but this method, applied to other refractory oxides, such as those of tantalum, titanium, zirconium, cerium, and aluminium, in the presence of tungsten, show that, in every case, except for ceria, the procedure would be satisfactory for the quantitative determination of small percentages of these oxides. Hence this method is not applicable to filaments of unknown composition without further operations on the residue. M. E. Nottage.

Patents.

Production of iron or other metals which combine with carbon, or alloys of such metals. H. G. E. Cornelius (F.P. 606,793, 23.11.25. Conv., 27.11.24).—Mixtures of finely-powdered ores containing oxides of the metals, with carbon, with or without the addition of ferrosilicon, ferromanganese, or aluminium, and a binding agent, are compressed into blocks, and heated electrically in a slag furnace. Heating may be by the arc or by electrical resistance, or by a combination of both methods, and the temperature is regulated by moving up and down the vertical electrodes. If the ore contains phosphorus or sulphur, the heating is repeated

in the presence of a slag consisting of lime, carbon, and, if necessary, fluorspar. L. A. COLES.

Ferro-chrome alloy. T. D. KELLY (E.P. 264,414, 4.9.26).—An alloy which is malleable when cast, and can be easily worked when cold, consists of 10—35% Cr, 7—25% Cu or Ni–Cu (copper being in such proportions that there is at least 7% in the finished alloy), and the remainder iron. The metals, or their granulated ores, are covered with a flux, consisting of (A) calcium or magnesium chloride, or (B) calcium or magnesium oxide mixed with sodium or ammonium chloride, combined with an equal quantity of carbon. The whole is melted at red heat by means of an electric arc. M. E. NOTTAGE.

Iron alloys having little tendency to rust. H. G. E. CORNELIUS (F.P. 607,108, 27.11.25. Conv., 29.11.24).—Such iron alloys contain at most 0·05% C, 0·25—8·99% Cr, and at most the customary or smaller proportions of manganese, silicon, phosphorus, and sulphur, and, if desired, nickel, copper, or other elements which reduce rusting. J. S. G. THOMAS.

Production of corrosion-resisting coatings on iron and steel. C. H. HUMPHRIES, Assr. to METALS PROTECTION CORP. (U.S.P. 1,615,585, 25.1.27. Appl., 25.2.26).—Composite coatings, consisting of a layer of a metal electropositive to iron and a superposed layer of chromium, are electroplated on the surface of iron or steel articles. M. E. NOTTAGE.

Corrosion-resisting metal. C. E. JONES and M. A. ATUESTA, Assrs. to GENERAL ELECTRIC CO. (U.S.P. 1,615,707, 25.1.27. Appl., 10.11.24).—The article of ferrous metal is coated with a film of cadmium, which, in turn, is covered with a film of tin. M. E. NOTTAGE.

Coating iron articles with a heat-conducting metal. A. STRASSER (F.P. 607,381, 20.11.25. Conv., 17.7.25).—The surface of the article is decarburised by treatment with a mixture of the fluorides of potassium, sodium, manganese, barium, and calcium, then coated with aluminium by dipping in a bath of the molten metal. If desired, a further coating of a more highly-conducting metal may also be applied. A. R. POWELL.

Colouring iron and steel brown. LANGBEIN-PFANHAUSER-WERKE A.-G. (F.P. 607,828, 10.12.25. Conv., 8.1.25).—The articles are first treated anodically in an electrolytic bath, then coated with a deposit of electrolytic iron which is subsequently oxidised. The coatings adhere well, and do not rust. A. R. POWELL.

Welding cast-iron. GEBR. BÖHLER & CO., A.-G. (F.P. 607,402, 3.12.25. Conv., 10.12.24).—A soldering stick containing 0·1—15% Al and 3—15% Si is employed in the autogenous or arc welding of cast-iron. J. S. G. THOMAS.

Fluxes used in the welding of aluminium and its alloys, magnesium and its alloys, and other metals and alloys. J. R. BOOER, W. R. J. BRITTEN, and DISTRICT CHEMICAL CO., LTD. (E.P. 264,238, 15.10.25).—The flux is made by fusing together halides of alkali and alkaline-earth metals in molecular proportions or simple multiples thereof. An activating agent, *e.g.*, potassium fluoride, for accelerating the removal of oxide from the metal to be welded may also be incorporated. M. E. NOTTAGE.

Process of treating aluminium dross and the like. J. G. G. FROST, Assr. to NATIONAL SMELTING CO. (U.S.P. 1,615,009, 18.1.27. Appl., 17.3.26).—Metallic aluminium may be recovered from hot aluminium dross containing finely-divided particles of molten aluminium by conveying the dross upwards in an inclined container and causing the particles of molten aluminium first to coagulate and then to flow down the container. M. E. NOTTAGE.

Electroplating with chromium. F. W. WÜRKER (F.P. 607,829, 10.12.25).—The electrolyte consists of a solution containing 300—400 g. of chromium trioxide per litre, together with small quantities of one or more alkaline-earth or heavy-metal salts or hydroxides which are soluble in the solution. A current density of 1·5 amp./cm.^{2} at 60° is employed in the deposition. The electrolyte has a good throwing power and yields bright, adherent deposits. A. R. POWELL.

[Precious metal] alloys. SOC. ANON. CAPLAIN SAINT-ANDRÉ (F.P. 607,428, 3.12.25).—An alloy suitable for use as platinum substitute may be made by melting in a graphite crucible in an electric furnace a mixture of 61·5% Au, 28·5% Pd, and 10% Pt. A. R. POWELL.

Extraction of precious metals from the slimes of copper refineries. N. BARABOSHKIN, and "TRUST URALKUPFER" (E.P. 263,642, 26.1.26).—Copper is first removed by adding the requisite quantity of commercial sulphuric acid, heating and stirring the mixture for 2 hrs. at under 250°, extracting with hot water, and filtering. The residue is then roasted at 500—900°, by which means the volatile impurities, such as compounds of selenium and tellurium, are removed. The roasted product is mixed with 30% by wt. of soda-ash and 10% of pyrolusite, or any other suitable flux, and fused, or these fluxes may be added before the roasting process. The liquid slag is removed, and the recovered metal refined by means of blowing or by using oxidising fluxes. The low-temperature roasting prevents loss of precious metals by volatilisation and the formation of "matte" (*i.e.*, an alloy of the metalloid impurities with silver), and since the copper is removed from the slime before roasting, filtration of the raw slime is unnecessary. M. E. NOTTAGE.

Production of a nickel-copper alloy. W. F. COCHRANE, Assr. to U.S. INDUSTRIAL ALCOHOL CO. (U.S.P. 1,614,878, 18.1.27. Appl., 28.8.23).—A cast nickel-copper alloy contains 20—50% Ni, and less than 5% each of lead, tin, and zinc. The exterior portions of the casing have a higher nickel content than the interior, and the alloy is in the form of solid solutions with no intercrystalline structure. M. E. NOTTAGE.

Extraction of metals [zinc or tin]. F. A. and G. GRUESSNER (G.P. 432,394, 21.1.25).—The ore and coking coal either mixed together or in alternate layers are packed tightly and heated in a muffle furnace or retort so that tar and other hydrocarbons are expelled. The resulting mass having a structure of coke is transferred to a closed furnace and heated, whereby the metal is produced. The process is applicable to the extraction of zinc from roasted blende, zinc oxide, or brass turnings, or of tin from its ores. J. S. G. THOMAS.

Treatment of tin-containing ores and metallurgical products. F. Krupp Grusonwerk A.-G. (E.P. 256,634, 5.8.26. Conv., 7.8.25).—Flue dust containing tin may be produced from compounds containing tin by the addition to the raw material of a solid substance containing sulphur, *e.g.*, pyrites, whereby the tin is converted into tin sulphide in the interior of the layer of material to be treated, and passes out as such. The atmosphere of the furnace may be an oxidising one, in which case the tin sulphide is again burned to oxide and the tin recovered from the oxide flue dust; or a neutral one, in which case the tin is recovered from tin sulphide in the flue dust. To prevent oxidation of the sulphur compound, a small quantity of a reducing agent, *e.g.*, fuel, is added. M. E. Nottage.

[Producing] single crystals of metals, *e.g.*, tungsten, which are fusible with difficulty. Ver. Glühlampen u. Elektricitäts-A.-G. (Austr. P. 103,904, 31.12.24).—The purified metallic oxide, more especially thorium nitrate containing tungstic oxide, is submitted successively to heat treatment in the presence of hydrogen in zones at 700°, 750°, 810°, 880°, and 950°. The resulting metallic powder consisting of grains of different sizes is pressed into rods, pre-sintered at about 1000°, and finally sintered at a temperature within 85° of its m.p. In a few minutes single crystals which can be cold-drawn to form bright helices suitable for use in discharge tubes are obtained. J. S. G. Thomas.

Precipitating metals on an incandescent body. N. V. Philips Gloeilampenfabr. (E.P. 249,067, 6.10.25. Conv., 14.3.25).—Metals of the first undergroup of the fourth column of the periodic system, *e.g.*, zirconium, may be precipitated on an incandescent body, *e.g.*, a tungsten wire, by heating the latter in an atmosphere containing the iodide of the metal to be precipitated. The wire is hermetically sealed into a vessel made of chrome iron, which can be evacuated. The metallic iodide, or the metal and iodine, is introduced, and the vessel heated at above the decomposition temperature of the iodide. By means of an electric current the wire is heated to incandescence, the maximum temperature being determined by either the m.p. of the precipitated metal or of the alloy formed by it with tungsten. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 239,235 [see B., 1925, 908]). M. E. Nottage.

Continuous production of lead oxides in the dry state. G. Shimadzu (E.P. 263,644, 27.1.26; cf. B., 1922, 372 A; 1924, 711; 1925, 714).—The apparatus consists of a rotatable drum mounted on hollow trunnions. The interior of one of these trunnions acts as a screw conveyor for feeding pieces of lead into the drum, and is also provided with a pipe through which air or other oxidising gas under pressure can be passed into the drum. This gas impinges upon the pieces of lead and forms a coating of oxide thereon, the oxidation being aided by the heat of the reaction itself and by the friction of the pieces against one another. This coating is removed as a very fine powder by the abrasion of the pieces, and is carried by the gas current into a chamber connected with the hollow trunnion at the other end of the drum, and provided with a set of vertical bars on which the particles impinge and are deposited. The suboxide powder so collected may be removed as such, or may be converted into litharge by the initial application of heat. A tower provided with filter bags of silk etc. collects the remaining powder from the issuing gas. M. E. Nottage.

Process for oxidising non-ferrous metals. G. E. Rohmer, Assr. to Andrews Lead Co., Inc. (U.S.P. 1,616,233, 1.2.27. Appl., 30.9.25).—The metal is allowed to flow by gravity, in the form of a thin, relatively wide-spread sheet, through and out from the heated chamber of a furnace containing oxygen. M. E. Nottage.

[Lead-bismuth] alloy. R. Sernissy (F.P. 607,661, 20.3.25).—An alloy containing 65% Pb, 30% Bi, and 5% Sn is characterised by great resistance to corrosion by chemical reagents. A. R. Powell.

Treatment of metal sheet or wire in a gas current. Eisenwerk A.-G. Rothau-Neudeck (F.P. 608,114, 19.12.25).—Metallic articles may be annealed or heat-treated in an atmosphere of any suitable gas by the use of a closed electric furnace forming part of the gas circulating system. The gas from the furnace passes through a cooler or heat-interchanger, and the cool gas is used for cooling the articles as they pass from the furnace. A. R. Powell.

Manufacture of alloy steels. B. D. Saklatwalla (U.S.P. 1,616,393, 1.2.27. Appl., 15.4.22).—See E.P. 196,260; B., 1924, 751.

Extraction of metals and their compounds from ores and impure materials. A. Pacz, Assr. to General Electric Co. (U.S.P. 1,614,149, 11.1.27. Appl., 14.12.23).—See E.P. 226,217; B., 1925, 597.

Process of conglomerating fine ores, flue dust, roasted pyrites, small coke, and the like. O. Kippe (U.S.P. 1,614,369, 11.1.27. Appl., 17.6.25. Conv., 28.6.24).—See E.P. 238,246; B., 1926, 754.

XI.—ELECTROTECHNICS.

Determination of moisture in cellulose. Schlumberger.—See V.

Oxidation of iron at high temperatures. Fedotéev and Petrenko.—See X.

Determination of thoria in tungsten filaments. Brophy and Van Brunt.—See X.

Patents.

Detector metal. F. Thuaud (F.P. 606,605, 28.2.25).—A ferrosilicon alloy for use as a radio-detector is composed of 80—96% Si, 0·02—0·6% Se, and traces of sulphur, phosphorus, and carbon. The ferrosilicon is twice melted, reduced with addition of pyrites or other material containing selenium, and poured. J. S. G. Thomas.

Electrical resistance grid. C. T. Evans (U.S.P. 1,614,920, 18.1.27. Appl., 14.8.22).—A grid composed of 3·50—7·75% Cr, 10—20% Ni, 0·20—1·00% C, 0·25—2·50% Mn, 0·15—2·50% Si, 0·05—0·35% Cu, and the remainder Fe. F. G. Crosse.

Permanent magnet. Paul Elektrik G.m.b.H. (G.P. 432,274, 17.6.24).—Ferromagnetic material com-

posed of a nickel-copper alloy containing 8% Cu is magnetised up to its magnetic transformation point.
J. S. G. THOMAS.

Electrolytic cell. R. HERMANEK (G.P. 432,815, 23.10.23).—The outer or bottom surfaces of cylindrical or prismatic aluminium electrodes employed in a cell are not subject to the action of the acid or alkaline electrolyte in which aluminium hydroxide is soluble, *e.g.*, chromic acid or acetic acid may be used.
J. S. G. THOMAS.

Electrolyte for accumulators. COMP. GÉN. D'ÉLECTRICITÉ (F.P. 607,735, 23.10.15. Conv., 21.12.24).—A warm solution of the sulphate of either an alkali metal or of magnesium, aluminium, or zinc, or of a double salt of these metals in sulphuric acid of the usual strength for accumulators is cooled, whereby a part of the solute is precipitated as fine crystals, the mass becoming gelatinous or pasty. A gelatinous mass is produced where large currents are to be employed, in order that the internal resistance of the accumulator may be kept low. J. S. G. THOMAS.

Filter diaphragm for electrolytic purposes. J. BILLITER, and SIEMENS & HALSKE A.-G. (F.P. 608,309, 22.12.25. Conv., 23.12.24).—Fibrous asbestos is mixed with a stable filler, *e.g.*, calcium fluoride or steatite, and a binder, *e.g.*, magnesium acetate, aluminium acetate, or titanium oxalate. J. S. G. THOMAS.

[Double] incandescence lamp. Y. SAKAKURA (E.P. 261,358, 27.10.26. Conv., 11.11.25).

Paper for electric cables (Austr. P. 103,613).—See V.

Metallic crystals for discharge tubes (Austr. P. 103,904).—See X.

Apparatus for testing bread making (E.P. 246,474).—See XIX.

XII.—FATS; OILS; WAXES.

Wheat oil. C. D. BALL, JUN. (Cereal Chem., 1926, **3**, 19—39).—The oils extracted from wheat embryo and wheat patent flour by means of ether are not identical. The wheat embryo oil has a higher saponification value, a higher iodine value, contains more unsaponifiable matter, and has a lower sp. gr. than the flour oil. The oil obtained from the embryo by pressure has a slightly higher iodine value than that prepared by extraction. A crystalline substance, m.p. 96—97°, separates from the flour oil, which is not found in the embryo oil. From its reactions it is probably a sitosterol ester. The embryo oil, on keeping for three years in a stoppered bottle in the dark, showed only a slight increase in acidity, and developed only a trace of rancidity. F. R. ENNOS.

Linseed oil with a positive Storch–Morawski [Liebermann] reaction. H. WOLFF (Chem. Umschau, 1927, **34**, 17—19).—Some linseed oils give a positive Storch–Morawski reaction owing to the presence of an unknown accessory substance in the unsaponifiable part of the oil, though phytosterol is not the cause of the reaction. It is pointed out that often in association with the seed is a wax-like substance the character of which depends on climatic conditions and on the nature of the soil. At present it is uncertain how this reactive substance reaches the linseed oil. Complete isolation of the compound has not yet been achieved.
H. M. LANGTON.

PATENTS.

Purification and regeneration of vegetable fatty oils. BAUDOUIN and BASSET (F. P. 606,914, 4.3.25).—The well-stirred oil is treated with a spray of concentrated sulphuric acid, excess of which is later neutralised with an alkali carbonate solution, stirring being continued until all the evolved carbon dioxide is eliminated. After settling, separation into oil, water, and alkali sulphate layers occurs. S. S. WOOLF.

Treatment of oleaginous seeds or oleaginous vegetable substances for the extraction of oil therefrom. G. KAMMERMANN (U.S.P. 1,616,319, 1.2.27. Appl., 1.2.24).—See E.P. 231,296; B., 1925, 411.

Process for extracting fatty acids from oils and fats. K. F. WILHELM (U.S.P. 1,616,292, 1.2.27. Appl., 7.7.24. Conv., 24.12.23).—See E.P. 226,767; B., 1925, 411.

Avoiding shock chill in separating stearine (E.P. 261,450).—See II.

Purification of fats, oils, etc. (G.P. 433,159).—See II.

Colouring of plastic materials (E.P. 242,274).—See VI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Action of sulphur dioxide and water on oils and varnishes. A. P. LAURIE (J. Oil Colour Chem. Assoc., 1927, **10**, 14—15).—Greyish-brown films produced on mural paintings (in the Royal Exchange, London) are shown to be due to condensation of acid moisture. Of various paint materials, subjected to the action of the vapour of a saturated aqueous solution of sulphur dioxide in an enclosed space, drying oils were found to be very susceptible to attack, resins were somewhat affected, whilst waxes and cellulose products are more or less resistant. S. S. WOOLF.

Cadmium colours and their suggested application to the paint industry. H. W. D. WARD (J. Oil Colour Chem. Assoc., 1927, **10**, 4—14).—An account of the history, preparation, and properties of the various cadmium pigments—sulphide, sulphide–carbonate mixtures, and sulphide–selenide mixtures being the most prominent. The latter comprise a series of bright colours ranging from orange-yellow to maroon-crimson. The recently developed "cadmopones," which are suitable for use as industrial pigments, consist of cadmium sulphide or sulphide–selenide mixtures co-precipitated with barium sulphate. S. S. WOOLF.

PATENTS.

Preparation of oils for paint media. CHEM. FABR. DR. J. WIERNIK & CO. A.-G. (G.P. 431,845, 7.5.22).—Drying oils are heated with inorganic or organic compounds that decompose into colloidal substances or themselves assume colloidal properties. S. S. WOOLF.

Water-glass paints. G. Kropfhammer (G.P. 431,777, 2.12.24. Addn. to 409,856).—A paint medium composed of hydrocarbon and/or fatty oils, mixed with water-glass and hydrogenated aromatic compounds, alone or in the form of soaps, is claimed. S. S. Woolf.

Stabilisation of luminous paints. A. Bamberger (G.P. 433,479, 14.5.25).—Luminous paints are stabilised by the addition of a solution of the unsaponifiable portion of resins. S. S. Woolf.

White colouring matter. L. P. Devaucelle (F.P. 606,959, 10.3.25).—Solutions of zinc sulphate are treated with barium carbonate or barium hydroxide, the mixture is filtered, and the residue ignited. Or dry crystallised zinc sulphate may be ground up with barium carbonate and the mixture ignited. In place of barium carbonate the residue of the decomposition of barium cyanide by steam may be employed. The colouring matter consists of an equimolecular mixture of barium sulphate and zinc oxide. A. Rayner.

Up-grading of mineral pigments. Wilhelm Winkler A.-G., and H. Stocker (G.P. 432,216, 17.6.24).—Mineral pigments are introduced in a finely-divided state into the waste-waters of dyeworks etc., the colouring matters contained in the water being precipitated on the pigments. The waste-waters are cleansed as far as possible before the addition of the pigments. A. Rayner.

Preparation of ink [from crude-tar extracts]. F. C. Bunge, and Forschungsinstitut f. Bergwerks- u. Sprengstoffchemie sow. verw. Geb. (G.P. 433,158, 6.3.25).—To 1 litre of the aqueous distillate obtained in tar distillation, boiled with 10 g. of gum arabic, 20 g. of ferrous sulphate are slowly added, followed by 6·6 c.c. of sulphuric acid in 60 c.c. of water. On cooling, 3 g. of aniline dye are added. S. S. Woolf.

Fixing pigments and dyes. I. G. Farbenind. A.-G., Assees. of H. Krzikalla and A. Schneevoigt (G.P. 433,152, 27.7.24).—Condensation products of carbamide and aldehydes in such solvents as ethylene chlorohydrin are used in conjunction with the usual printing media, to render pigments and dyestuffs fast to washing. S. S. Woolf.

Preparation of resinous condensation products from formaldehyde and tar- or crude tar-oils containing phenols. Comp. des Mines de Vicoigne, Noeux et Drocourt (F.P. 607,655, 19.3.25).—Tar oils containing phenols are condensed with formaldehyde in the presence or absence of such catalysts as sulphuric acid, sodium hydroxide, ammonia, ammonium chloride, benzylamine, etc., application of heat and/or pressure being optional. Resinous condensation products are separated from the "neutral oil residue" by steam distillation under reduced pressure. S. S. Woolf.

Cement for articles made from artificial resins (particularly phenol-formaldehyde condensation products). Dr. H. Traun & Söhne (G.P. 432,485, 24.2.23).—The use of an intermediate layer of hard rubber and a cement of artificial resin in soluble form is described. S. S. Woolf.

Dyestuff inks (G.P. 431,369).—See IV.

Lignone derivatives (E.P. 263,520).—See V.

Colouring of plastic materials (E.P. 242,274).—See VI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Samples [of rubber] prepared with *p*-nitrophenol. H. P. Stevens (Bull. Rubber Growers' Assoc., 1927, **9**, 45—47).—The addition of a small quantity of phenol together with *p*-nitrophenol to latex before coagulation yields a rubber resembling smoked sheet in appearance, and with a rate of vulcanisation slightly lower than when phenol is not added. Vulcanisation tests with air-dried sheet confirm the view that *p*-nitrophenol, by preventing maturation, is favourable to a more uniform rate of vulcanisation. D. F. Twiss.

Rubber lining for grinding mills. Rogers.—See I.

Rubber and corrosion problems. Fritz.—See X.

Patent.

Colouring of plastic materials (E.P. 242,274).—See VI.

XV.—LEATHER; GLUE.

Effect of relative humidity on the destruction of leather by acid. J. A. Wilson and E. J. Kern (Ind. Eng. Chem., 1927, **19**, 115—116).—Samples of leather containing free sulphuric acid were subjected to atmospheres of different relative humidities, respectively, for 46 days, and the moisture content and tensile strength determined for each humidity. The acid caused a greater loss in tensile strength as the humidity increased; thus 100% relative humidity caused a loss of 62·2—69·7%. The increasing destructive action at increasing relative humidities appears to be due to the greater water content of the acid-treated leathers. Lowering the water content of such leathers actually retards the rate of destruction. Leather is destroyed only by the free acid, and not by acid in chemical combination therewith. The greater the moisture content of the leather the greater is the hydrolysis of the leather-acid compound; thus, the amount of free acid per g. of leather increases although its concentration diminishes. Apparently the increase in quantity of free acid is responsible for the accelerated destructive action. D. Woodroffe.

Patents.

Treatment of hides and skins. L. Sheard (E.P. 264,302, 23.12.25).—Hides and skins are delimed and bated by drumming them with 2—5% of their weight of sodium sulphite crystals (or other agent) for ½—2 hrs. without any addition of water. A quantity of sulphuric acid, about one-fifth of the weight of sodium sulphite used, is diluted with ten times its volume of water and added in two or three portions so as to obviate the escape of sulphur dioxide from the drum. The drumming is continued for ½—1½ hrs. Other suitable sulphites and acids may be used. D. Woodroffe.

Manufacture of artificial horn. J. E. Boyet and A. Gueudré (E.P. 250,607, 9.4.26. Conv., 11.4.25).—Animal materials such as sinews and tendons are dried, compressed between suitable rollers, or pounded, or crushed to convert them into a fibrous material which can be carded. This material is swollen for 2—5 hrs. in a 2% sodium sulphite solution, or caustic soda, or for 3—10 min. in a 5% sulphuric acid solution, washed on a

sieve with water at 18—24°, hydraulically pressed in a mould to produce a homogeneous paste, and treated with an excess of a concentrated solution of salt highly soluble in water (*e.g.*, 1—2 hrs. with aluminium chloride solution *d* 1·21). The fibres thus partly dehydrated and made firm are rendered insoluble with formaldehyde solution, moulded under pressure, and dried. D. WOODROFFE.

Apparatus for transforming gelatinic colloids into globules or pearls. W. WACHTEL (U.S.P. 1,614,636, 18.1.27. Appl., 9.11.25. Conv., 19.5.25).—The colloid is formed into drops which fall into a cooling liquid contained in a vessel through which there is a continuous flow of liquid, and at the lower end of which is an outlet fitted with a lock which carries away the solidified globules without disturbing the liquid.
D. WOODROFFE.

XVI.—AGRICULTURE.

Decomposition of farmyard manure in soil and its utilisation by plants. J. KÖNIG (Mitt. deut. Landw.-Ges., 1926, 552, 571; Bied. Zentr., 1927, **56**, 18—21).—The decomposition of farmyard manure has been followed in soils of six different types, both uncropped and cropped with sugar beet and rye. About 75% of the carbon of the dung is oxidised during the first year after manuring; oxidation of the remainder is slow. Pentosans show a still more rapid decrease than the total carbon, whereas the decomposition of lignins is very much slower. There is a substantial loss of nitrogen soon after manuring. The phosphoric acid soluble in 1% citric acid solution changes little, but there is a gradual loss of potassium. The utilisation of the nutrient constituents of the dung by plants in the first two years amounts to about 30% of the nitrogen, 25% of the P_2O_5, and 55% of the K_2O. The results are in close agreement with those of Bach (cf. B., 1926, 640).
C. T. GIMINGHAM.

Stimulating action of metal salts on the germination of barley. J. NOELDECHEN (Kühn-Archiv, 1926, **9**, 264—309; Bied. Zentr., 1927, **56**, 28—31).—Experiments are recorded on the effect of soaking barley seed for 1 hr. in solutions of metal salts at various concentrations on the germination and yield of dry matter. Stimulation of germination was observed at certain concentrations with all solutions except those of copper salts. In this respect, sodium phosphate was most effective; then salts of mercury, silver, lead, and zinc; and, finally, salts of manganese and copper. Data are also given in regard to the concentrations which caused injury.
C. T. GIMINGHAM.

Phosphoric acid requirements of German arable soils. O. LEMMERMANN and W. JESSEN (Z. Pflanz. Düng., 1927, **B6**, 2—10; cf. Lemmermann and Wiessmann, B., 1926, 600).—The results of the fourth year of this series of field experiments are given. Of the soils tested, about 29% responded definitely to phosphatic manuring, 14% showed a slight response, and 57% showed no response. C. T. GIMINGHAM.

Evaluation of phosphoric acid in Rhenania phosphate. H. NIKLAS, A. STROBEL, and K. SCHARRER (Fortschr. Landw., 1926, **1**, 401; Bied. Zentr., 1927, **56**, 15—18).—Ammonium citrate (Peterman's solution) as a solvent for the determination of phosphoric acid in Rhenania phosphate is compared with citric acid. The figures are considered in relation to the results of a series of vegetation experiments with a variety of crops, in which applications of equivalent amounts of phosphoric acid (as determined by the two solvents) in the forms of superphosphate, basic slag, and Rhenania phosphate were given. It is concluded that the citric acid-soluble P_2O_5 in Rhenania phosphate is not inferior in its action to the water-soluble P_2O_5 in superphosphate, and is superior to the citric acid-soluble P_2O_5 in basic slag; and, further, that the percentage of P_2O_5 in Rhenania phosphate which is soluble in ammonium citrate solution is a measure of the physiological efficiency of the fertiliser. C. T. GIMINGHAM.

Amounts of [plant] nutrients in surface soils and subsoils and their importance in soil investigations. F. HONCAMP and F. STEINFATT (Z. Pflanz. Düng., 1927, **B6**, 21—27).—The authors have compared the amounts of "available" phosphoric acid and potassium in a number of surface soils of arable fields with the amounts in the corresponding subsoils. The determinations were made by the Neubauer seedling method. The percentages of available nutrients in the subsoils were nearly always less than in the surface soils, but the relationship between the two figures varied very greatly from soil to soil. No conclusion could be drawn from an examination of a surface soil as to the stock of nutrients in the subsoil. Further, the depth of the surface soil should be taken into account in laboratory and vegetation experiments. It is also shown that the proportion of P_2O_5 and K_2O soluble in 10% hydrochloric acid, which is taken up by seedlings, varies greatly in different soils, and is always greater in a surface soil than in the corresponding subsoil.
C. T. GIMINGHAM.

Standard spray mixtures. R. E. ANDREW and P. GORMAN (Connecticut Agric. Exp. Sta. Bull., 1926, [278], 491—507; Chem. Abstr., 1926, **20**, 3531).—Soluble arsenic may be determined by a modification of Ramberg's method. Lime–sulphur reacts with lead arsenate, giving increased soluble arsenic and decreased sulphur in solution; it reacts similarly with lead arsenate–nicotine sulphate (which do not interact), and with lead arsenate–casein–lime. Addition of nicotine sulphate to lead arsenate and casein–lime increases the soluble arsenic. Casein–lime increases the soluble arsenic content of lead arsenate. Different orders of mixing quadruple mixtures give different results. A. A. ELDRIDGE.

Cocoa by-products and their utilisation as fertiliser materials. G. P. WALTON and R. F. GARDINER (U.S. Dep. Agric. Bull., 1926, [1413], 1—44; Chem. Abstr., 1926, **20**, 3519).—More than 33% of the total nitrogen of both pressed cake and solvent-extracted cocoa is water-soluble, but the insoluble organic nitrogen is of inferior quality. The alkaloid-nitrogen is water-soluble, and may form 50—60% of the total water-soluble nitrogen. The materials are compared with castor pomace and cottonseed meal. Cacao shells contain less nitrogen (of low quality) and phosphate but more potash than by-product cocoa cake.
A. A. ELDRIDGE.

Toxicity of dipyridyls and certain other organic compounds as contact insecticides. C. H. Richardson and C. R. Smith (J. Agric. Res., 1926, **33**, 597—609; cf. Smith, A, 1924, i, 558).—A crude oil containing several dipyridyls proved highly toxic to various species of aphids and to the larvæ of certain beetles and moths. It was less toxic than nicotine to the aphids and to some of the species of larvæ, and more toxic to other species of larvæ. It was not injurious to plants infested with aphids at concentrations which killed the aphids. The 2 : 2′-, 3 : 3′-, 3 : 4′-, and 4 : 4′-dipyridyls, which were present in the crude oil used, were individually less toxic to *Aphis rumicis* than the oil itself, the 4 : 4-compound being the least toxic. Certain other compounds closely related to nicotine showed little toxicity compared with the dipyridyl oil. C. T. Gimingham.

Effect of time of irrigation on the production of crude protein in wheat. A. Kezer (Cereal Chem., 1926, **3**, 340—342).—The highest protein production in wheat occurred when irrigation was carried out at the period of tillering and jointing of the plants. Later irrigation was usually less efficacious. Better production of protein occurred when the whole of the irrigation water was applied at one time, than when distributed over the growing period of the plant. Irrigation at the "heading" period produced the highest wheat yield and the best quality grain, although the protein content was not the maximum. A. G. Pollard.

Effect of sodium nitrate on growth of wheat. Davidson and Schollenberger.—See XIX.

Antiscorbutic activity of milk etc. Kieferle and Zeiler.—See XIX.

Patents.

Manufacture of a nitrogenous fertiliser. G. H. Buchanan and P. W. Griffith, Assrs. to American Cyanamid Co. (U.S.P. 1,614,521, 18.1.27. Appl., 8.2.24). —Calcium cyanamide is treated so as to obtain a solution of free cyanamide, which is then treated with an acid, and the mixture heated and substantially neutralised with ammonia. G. W. Robinson.

Treatment of phosphatic materials. B. P. Hill, and Blaydon Manure and Alkali Co. (1877), Ltd. (E.P. 263,576, 26.10.25).—Phosphatic material in a finely-divided state is mixed with powdered coal or other fuel, and, if necessary, silica or silicious material, and injected or carried into the combustion chamber by the blast of primary or secondary air feeding the fuel burner, or is allowed to fall through the rising flame of such fuel. The phosphorus is volatilised as such or as oxide and, if necessary, air is admitted to oxidise it completely to phosphoric acid, which is precipitated electrostatically or absorbed in chemical reagents. Suitable means are provided for tapping off fluid or semi-fluid residues deposited in the combustion chamber. G. W. Robinson.

Production of fertiliser materials. G. Barsky and F. W. Freise, Assrs. to American Cyanamid Co. (U.S.P. 1,614,943, 18.1.27. Appl., 23.7.23).—1000 pts. of refractory phosphate rock are mixed with 1300 pts. by wt. of sulphuric acid (*d* 1·56). After 2—3 min., 150 pts. of calcium cyanamide are added, and the whole is discharged into a den, broken up as soon as the mass has hardened, and stored in heaps. G. W. Robinson.

Production of tobacco extracts of high nicotine content. W. Schacht (G.P. 432,921, 21.12.23).—Dry tobacco, either during or after powdering, is mixed with alkalis, alkaline earths, etc. The mixture is moistened with water, and extracted by diffusion. The extracts obtained contain nearly all the nicotine and tobacco oils, and may be used in the treatment of parasitic diseases of domestic animals. The residues contain pectins etc., which serve as fertilisers and plant insecticides. B. Fullman.

Preparation of protective agents for plants. I. Kreidl (Austr. P. 102,956, 22.3.24).—Hydrocarbons, hydrogenated or chlorinated hydrocarbons, or their derivatives, such as petroleum, tetra- or deca-hydronaphthalene, become, on treatment with solutions of hydrogenated phenols, emulsifiable with soaps, and may be diluted with water. *E.g.*, a product which may be sprayed and is readily emulsifiable with water is obtained on treating 5 g. of a 5% solution of soap in methyl*cyclo*hexanol with 100 g. of petroleum. B. Fullman.

Apparatus for pulverising chalk and similar substances [for fertiliser]. G. de Guillebon, Assee. of E., G., and M. Normand (E.P. 258,251, 2.9.26. Conv., 10.9.25).

Lignone derivatives (E.P. 263,520).—See V.

XVII.—SUGARS; STARCHES; GUMS.

Consideration of certain reactions of starches with special reference to enzyme hydrolysis. A. J. Hermano and O. S. Rask (Cereal Chem., 1926, **3**, 361—392).—Determination has been made of the viscosities and of the resistances to hydrolysis by malt diastase and pancreatic amylase of a number of starch pastes both in the raw and in the cooked or gelatinised state. These properties are dependent not only on the botanical origin of the starch, but also on other factors; *e.g.*, starches from the same variety of wheat give rise to pastes the resistance of which to enzymic hydrolysis varies with the locality in which the wheat was grown. W. O. Kermack.

Patent.

Production of starch forming a mucilage in cold water. Pfeifer & Schwandner G.m.b.H. (G.P. 432,961, 29.10.24).—Caustic alkali in dissolved form and in fine division is allowed to act on dry starch, a chemical change taking place without special drying and concentrating process. A. Rayner.

XVIII.—FERMENTATION INDUSTRIES.

Drying of hops. Institute of Brewing Research Scheme. Report of the fifth season's work at the experimental oast, 1925. A. H. Burgess (J. Inst. Brew., 1927, **33**, 58—66).—Experiments on the inter-relationship of time of drying, depth of loading, and air speed show that the deeper the load the greater is the benefit derived from high air speeds. The rate of drying is unaffected by burning sulphur under the hops. No further improvement in the colour is obtained with concentrations of sulphur dioxide greater than 1 oz./1000 cub. ft. of air. Sulphuring does not affect

the amounts of α- and β-resins. The rate of drying, amount of resins, and the value of the samples are the same with open- and closed-fire methods of drying. The value of the hops as judged by colour and aroma decreases as the temperature of drying is raised from 15° to 100°. The preservative value, as indicated by the resins, is unaffected by temperatures up to 90°. Biological tests show very little difference in the preservative values. Humidification of the air lengthens the time of drying and does not improve the colour, aroma, or texture. The final moisture content of hops should not exceed 17% at the time of packing. By keeping hops compressed during drying, the content of whole hops is improved and the time of drying is not affected. The antiseptic value of green hops after sterilisation by heat, when tested biologically, is greater than that of the same hops dried in the kiln. C. RANKEN.

Enclosed wort refrigeration. W. J. WATKINS (J. Inst. Brew., 1927, **33**, 67—75).—The experiments were carried out on a Paraflow refrigerator working in parallel with two vertical refrigerators, 6 ft. high. Although introduction of air at the hot end of the machine was necessary for the sufficient aeration of the wort, no difficulty was experienced in obtaining absolutely sterile wort. Without aeration the worts flocculated equally as well as those cooled on the vertical refrigerators, whilst with aeration superior flocculation was obtained with the Paraflow machine, and the worts entered the fermentation vessels nearly bright, most of the flocculated matter being left behind on the cooling surfaces. The improved flocculation was attributed to the greater surface caused by agitation during aeration and partly to the greater length of flow of the wort while it was cooling through the critical stage, 39—21°. Larger amounts of amorphous coagulum appeared on the surface of the worts during the early hours of fermentation as a result of aeration in the Paraflow. C. RANKEN.

Surface of yeast as a factor in fermentation. C. RANKEN (J. Inst. Brew., 1927, **33**, 76—84).—The various substances in suspension in wort, into which yeast is seeded, coat the yeast and produce effects according to the specific nature of the coating substance. Calcium oxalate deposited on yeast greatly accelerates the fermentation by preventing the yeast from cohering and by causing it to remain almost completely submerged in the fermenting liquid, whilst yeasts coated with peptone–tannin retard the fermentation rate and give rise to immense compact yeast heads. Coatings of calcium phosphate and hop resins have intermediate effects. The effect of a composite coating is not the strict average of the individual deposits, calcium oxalate having a preponderating influence. The quantity of yeast in contact with the fermenting liquid and, also, the area of the surface of the fermenting liquid in contact with the air are governing factors in the rate of fermentation. The hydrogen-ion concentration of the final fermented liquid is affected by the coating of calcium phosphate, but by none of the other coatings tested. C. RANKEN.

Physico-chemical investigation of wines. L. CASALE (Staz. Sperim. Agrar. Ital., 1926, **59**, 143—206; Chem. Zentr., 1926, II, 1907).—Although an acid mixture can be prepared of equal strength, active acidity, and alkalinity to that of a wine, yet the p_H values differ owing to the action of the colloids in the natural product. A cloudy must has a greater alkalinity and a higher p_H than clear must. Clear must, boiled so as to coagulate the colloids and brought back to the original volume after filtration, has a greater alkalinity and a higher ash content than the original liquid. The colloids adsorb the cations, thus affecting the p_H, the degree of adsorbtion depending on the ionic concentration. In wines of higher active acidity a larger proportion of hydrogen ions is adsorbed, whereas in wines with a high content of salts more cations are adsorbed. The titration acidity, with litmus as indicator, of wines with added calcium sulphate or sulphuric acid does not agree with the theoretical value, a part of the SO_4' and PO_4' being adsorbed by the colloids. Although the conductivity of an artificial mixture of acids alters according to known rules on the addition of alkali, the same treatment of natural wines gives abnormal results. With partially neutralised wine, the p_H is diminished, the concentration of cations increased, and a decrease of conductivity occurs due to interchange between the hydrogen ions adsorbed by the colloids and the cations in solution. The conductivity suddenly rises after complete neutralisation. C. RANKEN.

Detection of the adulteration of wine with extract of figs. F. GUERRIERI (Staz. Sperim. Agrar. Ital., 1926, **59**, 229—241; Chem. Zentr., 1926, II, 1908).—A 2% solution of aluminium acetate is mixed with wine or must freed from sulphuric acid. With unadulterated wine, the mixture remains clear for a considerable period, whereas wine adulterated with fig extract gives at once a gelatinous precipitate which gradually settles to the bottom of the containing vessel. By working with graduated vessels and allowing it to settle for 24 hrs. the test may be made quantitative. C. RANKEN.

Factors of bread production. HARREL.—See XIX.

Dough for bread making. SWANSON and WORKING.—See XIX.

Effect of yeast fermentation on flour. SHARP and SCHREINER.—See XIX.

PATENTS.

Production of dehydrated alcohol. E. A. BARBET (E.P. 264,050, 26.4.26. Addn. to E.P. 224,921; B., 1925, 608).—The crude alcoholic vapours condensed in the heater of the vacuum rectifier are pumped to the purifier, the purified alcoholic liquid being in turn drawn in by the vacuum of the rectifier, the absolute alcohol automatically delivered into a test safe by gravitation, and the head or tail products extracted by pumps replacing the vacuum vessels. If cool water is not procurable, the alcohol obtained between the azeotropic point and 100° is rectified in an additional hyperazeotropic column which works at atmospheric pressure and is heated by a tubular heater. The absolute alcohol is extracted at the base while the upper alcohol at the azeotropic point returns into the large rectifier under vacuum. C. RANKEN.

Distillation of alcohol. E. I. CLAPP, Assr. to U.S. INDUSTRIAL ALCOHOL CO. (U.S.P. 1,614,877, 18.1.27.

Appl., 10.1.25).—An alcoholic aqueous liquid containing lower-boiling impurities is distilled, and the alcohol and alcohol vapour at a higher concentration than the liquid being distilled are conveyed to a column for redistillation in order to remove the lower-boiling impurities.
C. Ranken.

Food product (E.P. 264,064).—See XIX.

Bread making (E.P. 246,474).—See XIX.

XIX.—FOODS.

Some variable factors of bread production. C. G. Harrel (Cereal Chem., 1926, 3, 1—18).—The influence of the following factors on the quality of bread is discussed:—correct panning, pan greasing, time and speed of mixing, temperature of the finished dough, absorption, fermentation, and the action of acids on the dough. The quality of the bread is shown to be dependent on the time of fermentation in straight doughs, but in sponge doughs it is independent of the length of the sponge period, being governed by the duration of the final dough period. The action of acids in bread production is influenced by two opposing factors: the one, the buffer value of the dough ingredients, tends to resist any increase in acid formation and so to increase the period of fermentation; the other is the quantity of salts and yeast used, an increase in which has the effect of diminishing the time required for fermentation.
F. R. Ennos.

Wheat and flour studies. V. Plasticity of simple flour-in-water suspensions. P. F. Sharp (Cereal Chem., 1926, 3, 40—56).—An apparatus is described for measuring the rate of flow of fairly concentrated suspensions of flour in water under pressure, in which the volume of the suspension passing through a suitable capillary in unit time, when subjected to varying pressures, is determined. From the results obtained, the yield values, or pressures just sufficient to start the flow, were determined graphically. By plotting the yield values against composition and extrapolating to the composition axis, the composition of the flour-in-water suspension at which it ceased to be a true liquid and became a plastic solid was determined. The flour suspension examined was plastic when it contained 9% or more of flour by weight on the dry basis.
F. R. Ennos.

Wheat and flour studies. VI. Effect of yeast fermentation on the proteins of the flour. P. F. Sharp and O. M. Schreiner (Cereal Chem., 1926, 3, 90—101).—The progress of fermentation of a normal dough containing 4% of yeast was followed by removing portions at intervals and making determinations of total proteins, amino-acids, potassium sulphate-soluble and alcohol-soluble proteins. So far as this method of protein-solubility fractionation showed, the yeast appeared to produce no appreciable change in the proteins of the flour. Comparisons were also made of the yield values and consistencies of suspensions formed from dough mixtures with varying amounts of yeast, after removal of the electrolytes and treatment with lactic acid. These plasticity constants were found to increase as the fermentation progressed, the change in the yield value being more apparent. F. R. Ennos.

Wheat and flour studies. VII. Milling and baking tests of frozen and non-frozen wheat harvested at various stages of maturity. W. O. Whitcomb and P. F. Sharp (Cereal Chem., 1926, 3, 301—315).—Freezing of matured wheat kernels had no injurious effect on the baking qualities of the flour when the moisture content of the wheat was less than 46% at the time of freezing. Flour from immature frosted wheat had a poor baking value. The baking value of flour was improved by soaking the wheat in water and redrying before milling. A. G. Pollard.

Wheat and flour studies. VIII. Composition of wheat and mill products from frozen and non-frozen wheat harvested at various stages of maturity. P. F. Sharp (Cereal Chem., 1926, 3, 402—410).—Analyses have been made of the protein content and ash of wheat and mill products obtained from crops harvested at various stages of maturity and in some instances frosted in the field. W. O. Kermack.

Control of diastatic activity in wheat flour. I. Production of diastatic flour and effect of large dosages. R. C. Sherwood and C. H. Bailey (Cereal Chem., 1926, 3, 107—136).—The diastatic activity of mixtures of flour with varying proportions of germinated wheat was determined from their content of reducing sugars. The results showed that with 5% and 10% of wheat germinated for 3 days, the diastatic activity increased two- and three-fold, respectively, but larger percentages did not result in a corresponding increase. With wheat germinated for 5 days, the rate of increase in activity began to diminish after 5% was exceeded. A small quantity of wheat germinated for 5 days was thus much less desirable than two or four times that quantity germinated for 3 days. Baking tests made on mixtures containing up to 20% of germinated wheat showed a gradual increase in loaf volume and expansimeter volume, but in all cases the grain and texture of the bread suffered. Determinations of total nitrogen and water-soluble nitrogen showed a relatively greater increase in the latter as the percentage of germinated wheat increased, this being more marked in wheat germinated for the longer period. The progressive breaking down of the proteins probably accounted to a certain extent for the reduced baking value of the mixtures with larger proportions of germinated wheat.
F. R. Ennos.

Control of diastatic activity in wheat flour. II. Experiments with flour milled on a commercial scale. R. C. Sherwood and C. H. Bailey (Cereal Chem., 1926, 3, 163—182).—Wheats low in diastatic power may have this property, together with the consequent baking strength of the flour, augmented by the addition of sprouted wheat. To obtain optimum results with wheats which alone yield flours with a diastatic power of about 100, the addition of between 3—5% of wheat germinated for 3 days is recommended. Addition of 2—3% of germinated wheat approximately doubles the rate of sugar production in doughs made from the resulting flours, but addition of more than 3% did not produce a corresponding increase. Milling yields are unaffected by the addition of 2—3% of wheat kernels germinated for 3 days. Improvement in baking strength is

of the flours was evidenced by increased gas production, a small increase of gas retention, and superior grain and texture of the loaf. The presence of 2—3% of germinated kernels did not affect the ageing of the flour, its protein quality, or the protease activity.

D. G. HEWER.

Factors affecting the diastatic activity of wheat flour. C. E. MANGELS (Cereal Chem., 1926, 3, 316—322).—The diastatic properties of several varieties of wheat flours are described. Results obtained indicate that the diastatic power of any particular variety of wheat may be affected by changes in climatic conditions, system of cropping, and fertiliser treatment. Variations in diastatic properties of flours are ascribed to the susceptibility of the starch granule to diastase attack rather than to the amount of enzyme present.

A. G. POLLARD.

Effect of sodium nitrate applied at different stages of growth of wheat on the baking quality of the flour. J. DAVIDSON and J. H. SCHOLLENBERGER (Cereal Chem., 1926, 3, 137—143).—Applications of sodium nitrate to growing wheat appear to be most effective at or just before heading time, and the flour, bran, and short all show a nitrogen increase, the first two about 30% and the last 23% over controls. The yield of flour is much as usual, and the wheat plump, whilst the resulting bread is distinctly superior in quality.

D. G. HEWER.

Relation of protein content to baking quality of flour from hard red spring and durum wheats. C. E. MANGELS (Cereal Chem., 1926, 3, 150—157).—Whilst good baking properties were found to be positively related to high protein content for 8 out of 11 crop years for hard red spring wheats, the quality of the gluten and the diastatic activity of the flour were also concerned. In the case of durum wheat flours the loaf volume was consistently smaller than with the hard red spring flours (evidently owing to poor gluten quality), and a positive correlation was found between protein content and baking strength.

D. G. HEWER.

Rapid electrometric method for the measurement of hydrogen-ion concentration of flour-water suspensions. H. J. DENHAM and G. W. S. BLAIR (Cereal Chem., 1926, 3, 158—162).—The quinhydrone electrode is a simple and robust form suitable for work on flour extracts. If flour and water mixtures are used directly there is a tendency for a continuous potential drift, and a preliminary extraction is preferred whereby 20 g. of flour are mixed with 200 c.c. of "conductivity" water in a borosilicate flask tested for buffer action, and, after 5 min. shaking, poured into 10 c.c. tubes and centrifuged for 5 min., the clear supernatant liquid being used in the electrode vessel.

D. G. HEWER.

Rapid electrometric method for the measurement of hydrogen-ion concentration of flour-water suspensions. H. J. DENHAM and G. W. S. BLAIR (Cereal Chem., 1926, 3, 427).—A new curve is given for the conversion of quinhydrone electrode potentials to p_H values to be used in the place of the incorrect curve previously published (cf. preceding abstract).

W. O. KERMACK.

Leavening agents for self-rising flour. P. LOGUE and I. T. RANKER (Cereal Chem., 1926, 3, 325—340).—A method for conducting baking tests of self-rising flour used for biscuit making is described. Baking values are partially dependent on the amount of water used in making the dough and upon the extent to which the dough is kneaded. The reaction of the leavening mixture necessary for optimum baking results differs with different flours. The p_H value of the finished biscuit is of value in determining the requisite leavening mixture.

A. G. POLLARD.

Antiscorbutic activity of milk from cows receiving silage, its biological value in comparison with milk produced by feeding dry fodder and brewers' grains, and its suitability for children. F. KIEFERLE and K. ZEILER (Fortschr. Landw. 1926, 1, 83; Bied. Zentr., 1927, 56, 37—39).—In experiments with guinea-pigs, cow's milk, produced by feeding silage, induced better growth and showed greater antiscorbutic power than milk from dry fodder (hay); and its superiority in both respects was still more marked over milk from brewers' grains. The high biological value of milk from silage and its special suitability for children are discussed.

C. T. GIMINGHAM.

Calculation of the value of foodstuffs for milk production and for fattening stock. N. HANSSON (Landw. Versuchs-Stat., 1926, 105, 1—15). G. FINGERLING (*ibid.*, 16—22).—The literature concerning the starch equivalent of proteins in foodstuffs is reviewed and discussed from the point of view of the difference between the Swedish figure (1·43) based on milk production records, and the Kellner value (0·94). The net energy value of foods is greater when used for milk production than for flesh formation. Experimental work shows that this difference is least marked in bulky foods of low digestible protein content, *e.g.*, roots and straw. In reply, Fingerling states that it is impossible to express the increased value of foodstuffs for milk production compared with flesh formation by increasing the Kellner value for the starch equivalent of proteins to 1·43 (the ratio of calorific values). During digestion carbohydrates may lose as much as 10% of their energy values by the formation of methane. Proteins may also suffer energy losses up to 18·7%. These losses are variable, and depend upon the composition of the foodstuffs and the kind of animal utilising them.

A. G. POLLARD.

Wheat oil. BALL.—See XII.

Effect of irrigation on protein in wheat. KEZER.—See XVI.

Cocoa by-products as fertilisers. WALTON and GARDINER.—See XVI.

PATENTS.

Process and apparatus for testing the different steps in bread-making. H. DEUTSCH-RENNER (E.P. 246,474, 20.1.26. Conv., 20.1.25).—An apparatus is described for obtaining a continuous record of the electrical conductivity of the dough during each stage of bread-making. By comparison with the values obtained during the correct making of bread from the mixture concerned, the progress and accuracy of each operation, as well as the time required, are determined.

F. R. ENNOS.

Heat-treatment of cereal substances. C. W. CHITTY, D. W. KENT-JONES, and WOODLANDS, LTD. (E.P. 263,897, 24.7.25. Cf. E.P. 228,829; B., 1925, 296).—A wheaten cereal, preferably in the form of flour, is dry-heated for a considerable period beyond that sufficient to render the gluten non-retainable by a washing test (a suitable heat-treatment is 7 hrs. at 76—93°). In this way a much smaller proportion (1% or less) of heated flour is needed for mixing with ordinary flour to increase the "strength" sufficiently. F. R. ENNOS.

Manufacture of a food and the product thereof. A. E. WHITE. From NORTHWESTERN YEAST Co. (E.P. 264,064, 1.6.26).—Cereal flour is gelatinised with water at 85° and treated with an extract of barley malt, the temperature being maintained at about 68° until the mixture gives no starch reaction. Yeast is then added and allowed to grow in and on the material for 4 hrs. at 32°, and finally, after the addition of milk, the whole is dried at a sufficiently high temperature to destroy the fermenting power of the yeast, and powdered. F. R. ENNOS.

Artificial drying of crops. B. J. OWEN (E.P. 263,595, 20.11.25. Addn. to E.P. 235,273; B., 1925, 648).—Structural alterations to the chamber ensure a more uniform penetration of the hot air from the central space. G. W. ROBINSON.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Testing of aspirin and acetylsalicylic acid and a note on "Togal." H. VALENTIN and A. LIEBER (Apoth.-Ztg., **41**, 567—568; Chem. Zentr., 1926, II, 2001).—Free salicylic acid is shown by the presence of reddish-violet coloured particles when aspirin or acetylsalicylic acid is moistened with 2% ferric chloride solution on a microscope slide. Tablets of both substances containing 20% of starch showed a faint violet coloration when thus tested, while the pure substances gave negative results. A determination by means of ether extraction is only approximate, as decomposition sets in on evaporation. The lithium content of "Togal" is only half that indicated by the manufacturers. B. FULLMAN.

PATENTS.

Preparing urea [carbamide]. J. M. A. STILLESEN (U.S.P. 1,614,698, 18.1.27. Appl., 16.6.23).—Calcium cyanamide is treated with water, a substantial proportion being converted into dicyanodiamide, which is separated and converted into carbamide by heating it with water in the presence of carbon dioxide. B. FULLMAN.

Preparation of soluble organic antimony compounds. CHEM. FABR. VON HEYDEN A.-G., Assees. of H. SCHMIDT (G.P. 424,952, 18.4.23. Addn. to 423,133).—Dihydroxynaphthalenesulphonic acids or their alkali salts are treated with antimony trioxide or hydroxide; *e.g.*, a solution of sodium ammonium 1:2-dihydroxynaphthalene-3:6-disulphonate is treated with antimony trioxide and sufficient sodium hydroxide to ensure neutrality to the end of the reaction. On filtration and concentration, a greenish, soluble, complex *compound* is obtained. The products have therapeutic application. B. FULLMAN.

Medical preparation of colloidal bismuth. V. A. LAPENTA and S. REISLER (U.S.P. 1,615,226, 25.1.27. Appl., 15.2.26).—A colloidal bismuth product maintaining its properties at high concentration is obtained by a process of reduction utilising the combined action of formaldehyde and dextrose in alkaline medium. The product is stabilised by the presence of a colloidal polymeride formed from dextrose by the action of formaldehyde and alkali. B. FULLMAN.

1-Methoxymethyl-3:7-dimethylxanthine. K. SCHRANZ and C. LUTTER, Assrs. to WINTHROP CHEMICAL Co., INC. (U.S.P. 1,616,282, 1.2.27. Appl., 6.7.25. Conv., 29.10.24).—See E.P. 242,296; B., 1926, 216.

Preparation of solutions of derivatives of diaminodihydroxyarsenobenzene. S. R. MACEWEN (U.S.P. 1,615,989, 1.2.27. Appl., 22.10.23).—See E.P. 221,565; B., 1924, 966.

Manufacture of complex metallic arsenobenzene compounds. K. STREITWOLF and J. HALLENSLEBEN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,616,204, 1.2.27. Appl., 19.3.24. Conv., 9.4.23).—See E.P. 214,237; B., 1925, 691.

Process of making pharmaceutical product containing arsenic. W. SCHOELLER and M. GEHRKE, Assrs. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,616,144, 1.2.27. Appl., 22.6.25. Conv., 2.7.24).—See E.P. 236,563; B., 1925, 783.

Anthelmintic. H. WEYLAND, Assr. to WINTHROP CHEMICAL Co., INC. (U.S.P. 1,616,291, 1.2.27. Appl., 2.9.25. Conv., 21.11.24).—See E.P. 243,325; B., 1927, 29.

Tobacco extracts (G.P. 432,921).—See XVI.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Duplex photographic emulsion. H. J. MALLABAR (Brit. J. Phot., 1927, **74**, 29—30).—A formula and instructions are given for the manufacture of a gaslight emulsion containing hydroxylamine. Development is by immersion in caustic soda solution. The emulsion is also suitable for print-out papers, and, by addition of copper sulphate during manufacture, it is rendered extremely vigorous as a gaslight emulsion. The coated material keeps better than ordinary papers, and is unaffected by impurities in the paper base. W. CLARK.

New hyper-sensitising methods for autochrome plates and others. A. NINCK (Brit. J. Phot., 1927, **74**, Col. Suppl., **21**, 5—7. From Le Photographe).—Satisfactory formulæ are given for hyper-sensitising baths containing ammoniacal silver nitrate together with: (*a*) Pinachrome + Pinacyanol; (*b*) Pinachrome + Pinachrome Violet; (*c*) Pinachrome + Pinacyanol + Pinachrome Violet, and (*d*) Pinaverdol + Pinachrome Violet. The keeping qualities of hyper-sensitised autochromes are discussed. The best keeping qualities result if the plates are washed in water followed by alcohol-water baths after hyper-sensitising. The baths recommended for autochromes can be used for hyper-sensitising ordinary plates, the Pinachrome–Pinachrome Violet formula being preferred. W. CLARK.

Sensitivity of films free from silver [dichromated gelatin]. O. MENTE (Phot. Ind., 1926, 1035—1036).—The author tests the claims of Westland Film G.m.b.H. (G.P. 427,082) that addition of ferricyanide and bromide to a 3% dichromate solution results in a great increase of sensitivity of gelatin bathed in the solution. Using the formula given in the patent, the sensitivity obtained was identical with that found with the plain dichromate solution. W. CLARK.

Photographic measurement of rate of detonation of explosives. PERROTT and GAWTHROP.—See XXII.

Photographic studies of explosives. URBÁNSKI.—See XXII.

PATENTS.

Production of fadeless materials (E.P. 264,021).—See V.

XXII.—EXPLOSIVES; MATCHES.

Determination of alcohol in dehydrated [nitro-] cotton. CHENEL (Mém. Poudres, 1926, **22**, 143—144).—Alcohol may be determined in nitrocotton, in which water has been displaced by alcohol, by placing 7—10 g. of it in one limb of a U-tube which is immersed in a glycerin bath at 50°, and then aspirating over it, at the rate of 1 litre/hr., air which has passed over pumice soaked with sulphuric acid and pumice soaked with caustic potash. The air after passing over the nitrocotton passes over potassium carbonate and then over pumice moistened with sulphuric acid. By a series of weighings the amount and strength of the alcohol in the nitrocotton may be obtained. S. BINNING.

Photographic measurement of rate of detonation of explosives. G. ST. J. PERROTT and D. B. GAWTHROP (J. Franklin Inst., 1927, **203**, 130—110).—The apparatus comprises a horizontally-rotated drum around which is wrapped a sensitised film, and a quartz lens which focusses the image of the flame of detonation through a slit on to the film. The drum is driven by a motor at a peripheral speed of 20 m./sec., and a tachometer is connected to the motor. Since the film moves horizontally and the explosive does not detonate at an infinite rate the image of the flame will be inclined at an angle depending on the rate of detonation, and this rate is calculated from this angular measurement. Various explosives have been tested, and the results are essentially the same as with the Mettegang recorder. W. G. CAREY.

Photographic studies on the detonation of explosives. T. URBÁNSKI (Rocz. Chem., 1926, **6**, 838—847).—Photographs of the explosion of ammonal, bradite, T.N.T., dynamite, and nitroglycerin in glass tubes exhibit a number of dark spaces and bands in the flame, explained as being the seat of endothermic, secondary reactions. In some cases these bands are arranged helically, pointing to the helical propagation of the explosion wave, together with the products of explosion. R. TRUSZKOWSKI.

Instability of bleaching powder etc. KAST and METZ.—See VII.

PATENTS.

High explosive composition. W. R. SWINT, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,609,221, 30.11.26. Appl., 13.4.22. Cf. U.S.P. 1,594,861; B., 1926, 854).—A non-gelatinised explosive composition contains a normally liquid high explosive, ammonium nitrate, and sufficient bagasse pith to reduce substantially the density. W. CLARK.

Bulk propellant powder. A. S. O'NEIL, Assr. to WESTERN CARTRIDGE CO. (U.S.P. 1,608,808, 30.11.26. Appl., 26.1.22).—The powder includes in its composition a nitro-cotton of high nitration and a deterrent material to reduce the rate of combustion of the powder. W. CLARK.

Explosive. T. L. DAVIS (U.S.P. 1,607,059, 16.11.26. Appl., 8.3.24).—The explosive contains trinitrophenyl-*n*-butylnitroamine as one of its constituents. S. BINNING.

Absorbent for liquid oxygen explosives. C. MOTT and H. W. DAHLBERG, Assrs. to PUROX CO. (U.S.P. 1,606,889, 16.11.26. Appl., 23.7.24).—An absorbent for liquid oxygen consists of a mixture of peat and molasses which, after being dried and comminuted, is mixed with finely-divided carbon. S. BINNING.

[Waterproofing of] matches. P. M. FORSYTH (E.P. 264,394, 7.7.26).—Matches are waterproofed by coating with a mixture of stearin and alkanet root. Suitable proportions are 18 pts. by vol. of stearin to 1 pt. of alkanet root. During dipping the temperature of the mixture should be 52°. S. BINNING.

Testing of detonators. L. WÖHLER (U.S.P. 1,615,606, 25.1.27. Appl., 8.4.25. Conv., 16.6.24).—The detonator is embedded in a cavity in the explosive contained in a holder which is then placed on the lead plate of a detonator-testing apparatus. After initiation of the detonator its priming action is judged by the effect on the lead plate. S. BINNING.

Manufacture of nitroglycerin–nitrocellulose powders. L. PARODI-DELFINO (U.S.P. 1,609,303, 7.12.26. Appl., 22.9.25. Conv., 18.11.24).—See E.P. 252,978; B., 1926, 693.

XXIII.—SANITATION; WATER PURIFICATION.

Chlorine studies and some observations on taste-producing substances in water, and the factors involved in treatment by the super- and de-chlorination method. N. J. HOWARD and R. E. THOMPSON (J. New Eng. Water Works Assoc., 1926, **40**, 276—296).—Tastes and odours in chlorinated water resulting from the presence of phenolic substances and those of unknown origin are briefly discussed, and reference is made to the more important and conclusive cases of tastes of this character occurring in Europe and America, and to the research work undertaken. At Toronto at the present time the whole domestic supply, which is derived from Lake Ontario, occasionally develops a "medicinal" taste after chlorination. The most intensive periods are concident with excessive pollution of the raw water, high free ammonia, and increased organic matter. The taste periods of the longest duration usually follow severe storms on the lake, and are probably due, in part, to the stirring up of the organic matters. Those occurring in time of calm are coincident with excessive pollution of the lake

water with sewage containing industrial waste. The authors are of the opinion that phenoloid substances, occurring either in the industrial waste discharges or resulting from the decomposition of putrescible matters, are responsible for the taste. Super-chlorination and de-chlorination treatment for the removal of the taste has been tried. Upwards of three to four times the usual doses of chlorine were added, and after a suitable contact period the excess chlorine was removed by means of sulphur dioxide. Experiments showed that a definite amount of chlorine and also time contact are necessary to destroy taste effectively. Large amounts of chlorine with short contact periods are ineffective, but the time contact may be lessened by increasing the dose of chlorine. The contact period required depends upon the amount and character of the taste-producing substance present. Large-scale experiments using 6—10 lb. of chlorine per million gallons (Imp.) of water and allowing 1—1½ hrs. contact were completely and uniformly successful. It was noticed that for complete removal of the excess chlorine a definite excess of sulphur dioxide (about 25% in excess of the theoretical) was needed, probably on account of interaction of sulphuric acid formed in the water and the alkaline substances present in solution. Recent experiments indicate that taste may be influenced by the hydrogen-ion concentration of the water, acid and highly alkaline waters being least liable to taste, and have also shown that sunlight interferes with the colour production in the *o*-tolidine method for the determination of residual chlorine, but not to any great extent with the rate of chlorine absorption by the water. W. T. Lockett.

Physical and chemical conditions in the sea-water of the Zoological Society's aquarium. F. P. Stowell (Proc. Zool. Soc., 1925, 1241—1258).—The sea-water in circulation in the aquarium is kept in a fresh condition by a system of purification which comprises aeration, filtration, and storage in the absence of light. Comparative analyses of samples of the aquarium sea-water after being in use for 18 months, and samples of sea-water freshly drawn from the Bay of Biscay—the original source of the aquarium water—show that, with certain unimportant exceptions, the two waters are practically identical. Notable exceptions are the nitrate, phosphate, and silicate contents, which are, respectively, for the aquarium water, 5·0 pts./100,000, 2·56 mg./litre, and 4·0 mg./litre, as compared with 0·073 pt./100,000, 0·03, and 0·2 mg./litre for the fresh sea-water. W. T. Lockett.

Purification of sea-water by storage. F. P. Stowell (Proc. Zool. Soc., 1926, 245—255).—Fresh sea-water stored in the absence of light undergoes very slight change as regards hydrogen-ion concentration, alkaline reserve, carbon dioxide, nitrite, and nitrate content, whilst purification, as indicated by the free and albuminoid ammonia, proceeds steadily. With storage and exposure to sunlight, owing to photosynthesis and abstraction of carbon dioxide, the sea-water becomes excessively alkaline, the ammonia content increases considerably, and nitrates are reduced to nitrites. Samples of the sea-water in circulation at the Zoological Society's aquarium when subjected to dark storage and storage with exposure to sunlight gave results similar to those obtained with fresh sea-water. The experiments all indicated that the aquarium water is quite normal and comparable with the water of the open sea, and that the present system of dark storage is a safe method of eliminating excess bacteria etc., without harmful physical and chemical effects to the medium. W. T. Lockett.

Comparative studies of standard methods and the brilliant-green-bile medium on Lake Michigan water at Chicago. C. C. Ruchhoft (J. Amer. Water Works Assoc., 1926, **16**, 778—785).—Although brilliant-green–lactose–bile medium is much more specific for the colon group than standard lactose broth, the medium is entirely too inhibitory to some members of the colon group to be used as a direct medium for determining *B. coli.* When testing raw waters, brilliant-green–lactose–bile is a useful medium for confirmatory purposes, for use after preliminary enrichment in lactose broth as suggested by Jordan (B., 1926, 302). Further study is needed, however, before its usefulness as a confirmatory medium for use on chlorinated tap water can be ascertained. W. T. Lockett.

Water purification. IV. Adsorption of neutral salts by Kambara earth. S. Oikawa (J. Biochem. [Japan], 1926, **6**, 117—128; Chem. Abstr., 1926, **20**, 3525).—Kambara earth adsorbs chloride and sulphate from various neutral salts; calcium is adsorbed sufficiently for the softening of water. In a mixture of salt and acid, adsorption of the former is hindered, whilst the greater adsorption of the latter is generally increased. A. A. Eldridge.

Physico-chemical processes involved in the removal of manganese from drinking water. I. Adsorption of bivalent manganese by manganese dioxide. J. Tillmans, P. Hirsch, and F. Häffner (Gas- u. Wasserfach, 1927, **70**, 25—30, 58—63).—A study has been made of the absorption of manganese by manganese dioxide from solutions of manganous sulphate, which contained also a carbonate–bicarbonate buffer mixture in order to maintain a definite hydrogen-ion concentration. Equilibrium is attained after about one hour's shaking. The absorption is reversible, the same equilibrium being reached from either side. The amount of absorption increases with rise of temperature. From considerations based on the variation of the free and combined carbon dioxide in the solutions and the influence of the hydrogen-ion concentration on the amount absorbed, it is concluded that the manganous ion is absorbed directly, an equivalent quantity of sulphuric acid being liberated. The amount absorbed for a constant p_H varies with the equilibrium concentration of the solution according to the Freundlich adsorption isotherm, whilst for a constant end concentration the amount absorbed is a linear function of the p_H value. The process is regarded as a type of adsorption in which the acidic character of the manganese dioxide plays a part. A. B. Manning.

Chemical tests for fæcal impurities in drinking water. R. Schmidt (Gas- u. Wasserfach, 1927, **70**, 77).—The usual chemical tests for fæcal impurities in drinking water have only a limited value. The indican test proposed by Jolles (B., 1921, 59 A) is much more definite, but in some cases the extreme dilution of the impurity

or the possible adsorption of indican during filtration of the water through the soil renders uncertain the value of a negative result with this test. Better suited to the purpose is the bacterio-chemical indole test as described by Gersbach (Zentr. Bakt., 1922, I., **88**, 145).

A. B. Manning.

Extreme lability of certain mineral waters. W. Kopaczewski and A. de M. Sarmento (Compt. rend., 1927, 184, 109—111).—The variations with time of the p_H value, degree of ionisation, titratable alkalinity, and iron content of a meso-saline cold water from Vidago, containing 5·33% and 1·13% of sodium and calcium bicarbonates respectively, have been studied. After 30 min. a sudden temporary increase in the degree of ionisation coincided with the appearance of opalescence; the iron content rose to a maximum and then fell during the period 2—4 hrs., while the water gradually turned a yellow milky colour, and a yellow sediment settled out after 6 hrs. The other variables showed no corresponding fluctuations. Since the iron is not detectable at first in the water, it is concluded that it is present in a complex form, resistant to acid, which subsequently is precipitated as a result of ionisation changes due to the external conditions.

J. Grant.

Determination of nitrogen compounds in industrial effluents. H. Uthe (Z. angew. Chem., 1926, **39**, 1554—1557).—Owing to the variety of forms in which nitrogen occurs in industrial effluents, great difficulty arises in the application of the usual methods of water analysis to its determination. The simple distillation for ammoniacal nitrogen gives erroneous results if volatile amines or albuminous compounds are present. "Cold distillation" by a current of air in presence of magnesium oxide may give low results unless very long continued. The formalin method is not applicable to all waters, whilst titration with sodium hypochlorite gives errors in the presence of many amines. In the Kjeldahl determination of total nitrogen, losses may easily occur if amines are present, and the formalin titration in place of distillation does not give a clear end-point. Nitrogen as nitrate and nitrite may be determined by oxidation with acid permanganate and titration with indigo. If cyanides or thiocyanates are present they must be removed by precipitation with silver sulphate before determination of the total organic nitrogen.

C. Irwin.

Effect of poisons on the larvæ of flies. K. Feist (Z. Unters. Lebensm., 1926, **52**, 466—469).—The most toxic substances to fly larvæ were those producing insoluble compounds with proteins (*e.g.*, mercuric chloride, lead acetate, etc.). Numerous chemical substances were examined. Cocaine and novocaine showed very marked toxic effects, resorcinol rather less, and methyl alcohol none at all.

A. G. Pollard.

Toxicity of dipyridyls. Richardson and Smith.—See XVI.

Patents.

Continuous softening of water by the use of zeolites. C. H. Nordell (U.S.P. 1,608,661, 30.11.26. Appl. 25.7.21).—The apparatus consists essentially of a tank loosely packed with zeolitic material. The water to be softened enters through a pipe at the bottom, passes up through a central tube with a Venturi section at its base, and thus draws zeolitic material to the top of the tank. Thence it falls to the bottom again, maintaining a continuous circulation of material round the tank. In the upper part of the tank the fresh zeolite softens the water, which is then drawn off by a side tap; in the middle part brine is introduced by a side pipe to regenerate the exhausted zeolite; and in the lower part the zeolite is washed free from brine by water from a lateral opening in the entrance nozzle. The brine and wash water, which are under automatic control, are finally discharged through the same outlet at the base of the middle part of the tank.

F. R. Ennos.

Process of regenerating zeolites. E. G. Duden, Assr. to W. B. Scaife & Sons Co. (U.S.P. 1,611,422, 21.12.26. Appl., 3.7.25).—A solution of sodium chloride is drawn directly into and downwards through the zeolite contained in a tank, without first removing raw water from the tank.

F. G. Crosse.

Filtration of liquids [water]. G. C. Lewis (E.P. 262,328, 11.5.26).—A filtering element is made by filling a container of ceramic material, such as clay, with activated carbon, and burning at about 1370° to render the container porous and to increase the activity of the carbon while avoiding its oxidation. A germicide such as potassium permanganate or a copper salt may be incorporated with the carbon.

H. Holmes.

Screening or filtering apparatus [for industrial water supply]. F. W. Brackett & Co., Ltd., and F. W. Brackett (E.P. 263,693, 29.7.26).—The perforated plates of a travelling band screen are carried by endless chains passing at their lower ends beneath curved guides. Each plate carries a bucket or trough, and is so curved that the buckets form a continuous seal with a curved floor concentric with the guides. To prevent the buckets from fouling the floor, should the chains stretch, rollers are provided on the chains to run on curved guide strips at each side of the floor. The band is deflected at the beginning of its downward travel into an inclined direction, and jets of water are directed through it to wash off the foreign matter. The band travels with a working clearance between fixed sealing strips projecting at right angles to it.

H. Holmes.

Germicide [composition]. J. W. Churchman, Assr. to National Aniline & Chemical Co., Inc. (U.S.P. 1,614,281, 11.1.27. Appl., 13.7.23).—A stable germicide composition consists of a basic triphenylmethane dye and a neutral acridine dye, the former having a selective bactericidal action against Gram-positive bacteria and the latter a similar action against Gram-negative bacteria.

A. J. Hall.

Purification of air containing carbon monoxide or other deleterious gases and intended for respiration. H. Guillemard (U.S.P. 1,611,524, 21.12.26. Appl., 14.5.20. Conv., 3.10.17).—An oxidising agent is used comprising the product resulting from the interaction of iodic anhydride and concentrated sulphuric acid.

F. G. Crosse.

Filter [for water supply]. C. P. Eisenhauer, Assr. to Duro Co. (U.S.P. 1,609,867, 7.12.26. Appl., 3.8.23).

// BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 1, 1927.

I.—GENERAL; PLANT; MACHINERY.

Patents.

Heat exchange apparatus. G. U. Morgan (E.P. 264,377, 17.5.26).—A heat exchanger for two liquids which is specially suitable (as one of a pair) for heating oil fuel, as it can be easily and completely taken apart for cleaning, is claimed. B. M. Venables.

Heat exchanger. J. Price, Assr. to Griscom-Russell Co. (U.S.P. 1,617,083, 8.2.27. Appl., 29.9.25). —Separate heat-transferring units are assembled one above the other, each comprising a pair of headers connected by tubes which are slightly bent. The headers are so held that the tubes bend when heated and remove scale. B. M. Venables.

Grinding, crushing, pulverising, mixing, and separating machines. D. J. Rees (E.P. 264,324, 20.1.26).—In a grinding mill in which a number of roll heads, balls, or like grinding members are rotated by a spider and pressed centrifugally against a fixed ring, the driving spider is formed with resilient arms. B. M. Venables.

Atomisation and desiccation of liquids or solutions. Nestlé and Anglo-Swiss Condensed Milk Co. (E.P. 257,893, 15.6.26. Conv., 5.9.25).—Liquids issuing from one jet are subjected to a stream of hot, atomised air from another jet, at right angles to it. The jets are under suitable pressure or control, and are contained in a chamber, one side of which has an opening in direct alinement with the atomising jet, through which the atomised liquid is carried into the evaporating and collecting chamber. F. R. Ennos.

Method of concentrating fluids. W. B. McLaughlin (U.S.P. 1,615,287, 25.1.27. Appl., 17.1.23). —The concentration is effected at low temperatures by heat interchange with a hotter fluid, the heat passing from a small surface in contact with the heating fluid to a large surface in contact with the fluid to be heated. H. Holmes.

Method and apparatus for distilling liquids. W. F. Downs (U.S.P. 1,616,079, 1.2.27. Appl., 24.12.21). —The liquid is contained in a chamber provided with a partition near one wall to form a passage opening into the chamber at its upper and lower ends. Heat is applied to this wall to induce an upward circulation of the liquid through the passage, and means are provided for maintaining the upper end of the passage and liquid level in the chamber at predetermined relative heights. H. Holmes.

Vacuum evaporator. E. Heller (G.P. 432,903, 4.3.24).—The apparatus is provided with an external heating unit connected with the evaporator by an ascension pipe. The upper part of the heating unit is at the same height as the floor of the container of the evaporator, and the length of the ascension pipe corresponds to the temperature difference between the heating unit and the evaporator. By this arrangement a constant circulation of the liquid is obtained. R. A. A. Taylor.

Columnar hollow shapes for filling absorption towers etc. A. Letschert (G.P. 433,055, 18.4.23).—These shapes have in their outside surfaces openings which, in cross-section to their major axes, form approximately equilateral triangles with rounded corners. They have openings like narrow slits, parallel to or laterally placed to the sides, and slits which become narrower towards the inside. R. A. A. Taylor.

Method for low-temperature cooling, liquefaction, and separation of gases. A. Seligmann (U.S.P. 1,615,597, 25.1.27. Appl., 3.3.24. Conv., 26.5.22).—The gases are passed through a countercurrent heat-exchanger at a speed which is greater upon starting the process than during its normal continuance. H. Holmes.

Air-cleaning machine. J. C. Hosch (U.S.P. 1,616,802, 8.2.27. Appl., 27.7.25).—The air is passed through a primary set of zig-zag parallel vanes which terminate in hook-shaped collecting pockets. Two similar sets of vanes are provided for continuing the zig-zag motion. B. M. Venables.

Manufacture of colloidal or the like materials. W. H. Bentley, W. M. Coates, and J. Riley & Sons, Ltd. (E.P. 263,670, 7.6.26).—Stable solutions of colloidal substances for horticultural sprays and other purposes where the presence of electrolytes is a disadvantage, are prepared by the interaction, in the presence of a protective colloid in aqueous solution, of substances of such a nature and in such quantity that the solution contains the desired colloid, while the reaction product other than water contains little or no electrolyte, or is insoluble, or sparingly soluble, or is little dissociated. The quantity of water also may be limited, so that a soluble jelly is produced. W. G. Carey.

Non-corrosive liquid for fluid systems. M. R. Sherbino, Assr. to Hydraulic Brake Co. (U.S.P. 1,616,670, 8.2.27. Appl., 19.3.26).—Castor oil is dissolved in alcohol and saponified. H. Royal-Dawson.

Means for effecting the chemical analysis of liquids. H. S. Hatfield (E.P. 264,237, 14.10.25).—The apparatus comprises means for mixing known volumes of two or more liquids and recording the attainment or non-attainment of the end-point. In the case of testing the hardness of water, a measured volume of water is transferred to a tilting reacting vessel, and to it is added, drop by drop, standard soap solution. Each

drop of soap solution actuates an electrical contact which controls a pawl in a mechanical recorder. The solution is kept in agitation by a stream of air bubbles, and when the frothing point is reached the reaction vessel will overturn and start a fresh cycle. Another method of operation is to take fixed quantities of both reacting liquids and to record the attainment or non-attainment of the end-point by means such as a hydrogen electrode or a beam of light passing through the liquid and impinging on a photo-electric cell, the light being cut off if the liquid is coloured by an indicator. B. M. VENABLES.

[Illumination] photometer. H. WADE. From N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 264,039, 24.3.26).—In an illumination photometer of the kind in which a semi-transparent screen is illuminated on one side by a standard source of light of definite candle power, a resistance body of positive temperature coefficient is connected in series with the standard lamp in order to maintain the current through the lamp approximately constant for values of the voltage of the battery in its fresh and exhausted conditions. J. S. G. THOMAS.

Preserving physiological and like specimens. J. BRUNNER and E. SCHEELE (E.P. 263,674, 17.6.26).—Insects, plants, amphibia, reptiles, fishes, crustacea, etc. are preserved by embedding them in a soft or fluid-like condensation or polymerisation product, which permeates them, and which is then hardened by heating. All artificial resins may be used, the condensation products of formaldehyde and phenol or carbamide being especially suitable for animal and vegetable bodies. B. FULLMAN.

[Bridge for] furnaces. E. CROWTHER (E.P. 264,560, 9.7.26).

[Adjustable frame for portable] mixing apparatus. C. S. SIMMONDS (E.P. 265,073, 6.8.26).

Distillation and like columns. C. STILL and A. KUHN (E.P. 264,735, 20.7.26).

Nozzles for filters. JEWELL EXPORT FILTER CO., Assees. of J. E. WILLIAMSON (E.P. 256,228, 26.7.26. Conv., 28.7.25).

Apparatus for classifying [air-borne] materials. HARDINGE CO., INC., Assees. of H. HARDINGE (E.P. 257,568, 26.1.26. Conv., 28.8.25).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Freeman multiple retort of the British Oil and Fuel Conservation, Ltd. (Dept. Sci. Ind. Res. Rept., 1926, 35 pp.).—The retort is of the vertical continuous type, externally heated, and consists of six cast-iron chambers, 18 in. deep, placed vertically above one another. The temperature in each chamber is about 100° higher than in the one immediately above. The coal passes down through the chambers in succession, and is continually turned over during carbonisation (cf. B., 1919, 317 A). As a result of a preliminary run some minor alterations of the system were found necessary—in particular the feed and discharge valves were modified, the gas offtakes altered so that they could be readily cleared if they became choked, and improvements made in the arrangement of the ploughs for conveying the coal across the revolving trays. Four tests were carried out with a non-caking slack ("Kirkby Top Hard"), one with a feebly caking coal ("Brynna"), and one was commenced with a moderately caking coal ("Washed Waterloo Slack"), but had to be abandoned because the retort choked. The maximum temperature in the first test was 566°, but in the others did not exceed 500°. The weight of coal treated in each run varied from 7·5 tons to 15·7 tons, and the duration of the tests from 2 to 3 days. Losses on the weight and thermal balances varied between 1·29 and 3·2% on the former and between 3·8 and 8·4% on the latter. The yields from the "Kirkby Top Hard" (at 500°) were 13·4 cwt. of coke, 1610 cub ft. of gas of about 900 B.Th.U. per cub. ft., and 20·2 gals. of tar per ton of coal as charged. The corresponding yields from "Brynna" coal were 14·7 cwt. of coke, 1720 cub. ft. of gas of 978 B.Th.U. per cub. ft., and 18·6 gals. of tar. The coke was in the form of breeze, but was readily combustible; it could either be further ground and used as pulverised fuel or briquetted. The yield of tar was high, representing 80% of that obtained in the Gray–King apparatus (B., 1921, 205 A). The gas was a typical low-temperature gas, and yielded on scrubbing about 1 gal. of spirit per ton of dry coal. The yield of ammonia was low. The following are the yields in gals. per ton of dry coal of refined products from the tar of Test 3, and may be taken as representative—motor spirit (to 170°) 0·99; naphtha (170—220°) 0·65; burning oil (220—280°) 1·69; gas oil (280—320°) 1·15; lubricating oil (320—360°) 1·46; crude tar acids, 5·17; bases 0·23; the pitch amounted to 39·3% by weight of the tar. Estimates are made of the labour and power required to operate the plant. A. B. MANNING.

Influence of the ash content of a coal on the calculated calorific value of the ash-free coal. R. STUMPER (Brennstoff-Chem., 1927, 8, 33—36).—The calorific value of ash-free coal, calculated from the observed calorific value of the raw coal, varies with the ash content of the coal, the variation being particularly marked when the ash content exceeds about 18%. Possible explanations are discussed, but the variations appear to be principally caused by heat effects due to chemical changes occurring in the ash. The calculated calorific value of the ash-free coal is appreciably lowered by adding above 18% of silica to the coal, and slightly (but not regularly) raised by adding to the coal further quantities of its own ash. W. T. K. BRAUNHOLTZ.

Reactivity of coke. L. NETTLENBUSCH (Brennstoff-Chem., 1927, 8, 37—41).—When semi-coke, produced at 550°, is subjected to higher temperatures, its reactivity, as determined by Bähr's method (cf. B., 1924, 160), decreases, the decrease being particularly marked for superheat temperatures exceeding 800°. This is due to deposition of graphitic carbon on the coke surface, owing to decomposition of methane. On the other hand, the reactivity is practically unaffected by subjecting coke for a prolonged period to the same temperature as that at which it was produced. The degree of decomposition of coal gas by heat depends on the

temperature, gas velocity and concentration, surface area of the reaction chamber, and nature (particularly content and composition of ash) of the coal carbonised. The physical form in which the carbon is deposited depends largely on the nature of the hot decomposing surface. The reactivity of coke is enhanced by the presence of manganese dioxide and iron, but depressed by alumina and, slightly, by silica. The introduction of steam during carbonisation is advisable, in that it greatly reduces the deposition of carbon on the coke, thus rendering the latter easily combustible, and also protects the gas from the catalytic action of the ash.

W. T. K. Braunholtz.

Experiences with American and other coals during the miners' strike, 1926. G. Weyman (Gas J., 1927, **177**, 191—193).—The large stocks of coal held by gas works at the commencement of the strike had suffered variable amounts of deterioration during storage, resulting in a poorer quality of gas and coke, and slower rates of carbonisation. The Landsale, drift, outcrop, and heap coals which were utilised showed a very great variation in quality. Few of the European coals used were suitable as gas coals. The Silesian coals were practically non-coking, and the quality of the gas was poor. The Westphalian and Czecho-Slovakian coals gave fine-grained hard cokes, but again the gas was poor in quality, and difficulties were encountered in handling the small wet coal supplied. The American coals were uniformly good for gas-making, high yields of good quality being obtained; the tar yields were also good. Results of tests (on a 1/1000-ton scale coal-testing plant) are tabulated for all the types of coal discussed.

A. B. Manning.

Basic calculations and processes, together with the economics, of the conversion of ethylene in coke-oven gas into alcohol. W. Gluud and G. Schneider (Gas- u. Wasserfach, 1927, **70**, 97—102).—The raw gas must be subjected to a preliminary purification and concentration, including removal of tar, cyanogen, hydrogen sulphide, ammonia, and benzol, followed by compression, removal of carbon dioxide, and intensive cooling by the Linde process or the like. In this way a gas containing 70% of ethylene, 20% of methane, and about 10% of other hydrocarbons may be obtained. This is passed through a preliminary scrubber containing concentrated sulphuric acid, in which undesirable hydrocarbons (*e.g.*, acetylene, butadiene, propylene, etc.) are absorbed, then over quicklime (to remove sulphur dioxide), and finally through the main scrubber in which the ethylene is absorbed in concentrated sulphuric acid containing 1% of silver sulphate. The acid from the preliminary scrubber is diluted to precipitate resins, and may be concentrated or used for ammonium sulphate manufacture. The acid from the main scrubber, in which about 80% is combined with ethylene, is freed from catalyst, diluted with water, and from this solution alcohol (of about 40% strength) is distilled. The residual sulphuric acid is treated in the same way as that from the preliminary scrubber, and the alcohol is concentrated up to about 90% strength. Details are given for carrying out the process in the laboratory and on the large scale, and a detailed balance sheet is appended.

W. T. K. Braunholtz.

Extended formolite analysis of crude petroleum oils. A. M. Nastukoff (Petroleum, 1926, **22**, 1349—1350).—The great absorptive capacity of the formolite for saturated hydrocarbons is utilised in the further analysis of crude oils. From the first formolite number, F_1, obtained in the usual manner, a second, F_2, is obtained by treatment with light petroleum, and a third, F_3, by subsequent treatment with chloroform; this last is the true formolite number. Further calculated values up to F_7 are employed. Differences between these values indicate the yield of solar and lubricating oils, the resin content, cyclic unsaturated hydrocarbons, etc. A table gives the respective values for eight Russian and one Pennsylvanian crude oil.

H. Moore.

Anti-knock materials. G. Grote (Petroleum, 1926, **22**, 1344—1347).—The results of Charch, Mack, and Boord are discussed (cf. B., 1926, 570). The efficiency of lead tetraethyl being taken at 100, a table of comparative efficiencies is given. Whilst compounds of quadrivalent lead show anti-detonating properties, those of bivalent lead, with some exceptions, have no action. Lead benzaldehyde, lead diphenyl dinitrate, tin bromide, ferric chloride, aluminium triethyl and chloride, and bismuth triphenyl dichloride have no anti-detonating action. The anti-detonating action of substances on an acetylene mixture in an explosion tube varied greatly from results obtained on the motor. A direct relation was shown between intensity of detonation and electric conductivity of cylinder gases. The most powerful anti-knock materials deposit finely-divided particles of the metallic constituent on heating, and such compounds as undergo complete oxidation on heating are inefficient. Detonation is a consequence of the electric charge, rather than of the conductivity of the gases.

H. Moore.

Determination of the sand content of machine grease. C. Bunge (Chem.-Ztg., 1927, **51**, 64).—10 g. of the grease are extracted with benzyl alcohol and finally with hot water, and the residue is dried and weighed. The presence of sand grains being established by touching the residue, heated to glowing point, with a glass rod, the residue is treated with hydrochloric acid, filtered, and the remaining residue, after identification as sand under the microscope, is weighed. Amounts of sand, varying from 0·009—0·057%, have been found in samples of grease examined. There was no direct relationship between the percentage of sand and the number of grains present, as determined by the microscope method.

H. M. Langton.

Patents.

Coal-washing apparatus. Flow Coal Washery Co., Ltd., A. Russell, and S. Austin (E.P. 262,890, 16.10.25).—The suspended material in the coal to be cleaned is carried in a stream of water along a trough in which, at intervals, there are slots communicating with shale-evacuating boxes. These consist of vertical conduits, tapering from top to bottom, but uniform in width throughout, with the slots extending across the troughs. A stream of water enters these boxes through rectangular openings in one side. Above the water inlet and immediately below the coal-inlet slot is an inverted V-shaped bridge which spreads the coal and facilitates the cleaning action of the ascending stream of

water. Shale falls through the water stream, and the partially washed coal is returned to the trough for further treatment. S. PEXTON.

Compressed fuel for heating and cooking purposes. A. WENIGER (Swiss P. 114,655, 17.2.25).—Carbonaceous material is finely ground and treated with alkali carbonates; it is then sprinkled with dilute mineral acid, mixed with binding material, heated, and compressed. Any type of brown coal can thus be converted into a fuel resembling anthracite, and of high combustibility. A. B. MANNING.

Pre-treatment of a binding material for solid fuels. E. B. G. BASCOU (G.P. 433,734, 26.3.24. Conv., 10.4.23).—A mixture of dehydrated pitch and crude petroleum pitch is granulated by pouring the molten material in fine streams into water. Briquettes made using this material as binder are resistant to moisture and hold well together in the fire. A. B. MANNING.

Centrifugal gas washer. HAGER & Co. G.M.B.H. (G.P. 433,181, 25.6.20).—The washer is horizontal, and the wash liquid is driven by the gas pressure in a thin layer along the wall of the vessel. The liquid is collected by a gutter and used again after being returned to a holder and spray diffuser. The condition of the wash liquid is regulated by control of the quantity in circulation. R. A. A. TAYLOR.

Removal of solid substances from gases. G. J. PRAT (F.P. 606,783, 23.11.25. Conv., 24.11.24).—The gas to be purified is caused to flow through a rotating cylinder with perforated walls through which the solid substances pass, when they are given a helical motion about the axis of the cylinder. R. A. A. TAYLOR.

Rich gases from fuels. KOHLENVEREDLUNG G.M.B.H. (F.P. 606,886, 26.11.25. Conv., 23.1.25).—The gases laden with tar vapours pass directly from the retort through a cracking plant, avoiding introduction of foreign gases. A. B. MANNING.

Determination of the substances causing luminosity in gases, vapours, or mixtures of gaseous substances. I. G. FARBENIND. A.-G., Assees. of R. ENGELHARDT and W. LOMMEL (G.P. 432,237, 24.8.24).—The gases, before and after removal of the luminous constituents, are consumed in a burner to which air or oxygen is led, the amount of oxygen necessary to render the flame non-luminous being determinable by means of a flow-meter. R. A. A. TAYLOR.

Saturation and recovery of gases and vapours by solid absorbents. SOC. DE RECHERCHES ET D'EXPLOITATIONS PÉTROLIFÈRES (F.P. 607,683, 23.3.25).—The absorbent (activated carbon), charged with the gases and vapours, is treated with superheated steam to drive off the absorbed substance. R. A. A. TAYLOR.

Improvement in the Edeleanu process. ALLGEM. GES. FÜR CHEM. IND. M.B.H. (G.P. 432,580, 27.9.25).—Loss of liquid sulphur dioxide is minimised by saturating the charge with gaseous sulphur dioxide before introducing it into the reaction apparatus. A. B. MANNING.

Extraction of petroleum from oil-sands, bitumen from oil-chalks, oil-shales, coals, etc. H. PRELLER (G.P. 433,978, 30.8.22. Addn. to 400,122).—The raw material is first washed by being passed counter-current to a stream of hot water, the coarser oil and mineral particles being thus separated. The finer particles still containing oil pass into a second washing vessel of considerably greater cross-section, where the more slowly moving stream permits separation of the fine oil particles. Loss of oil is thereby avoided. A. B. MANNING.

Continuous distillation of crude petroleum oils. F. M. ROGERS and R. E. WILSON, Assrs. to STANDARD OIL Co. (U.S.P. 1,615,407, 25.1.27. Appl., 11.10.24).—The oil is reduced in conventional stills to a very heavy residue; this is then passed with steam through a pipe still in which it is heated at 370—425°, thence to a separating chamber in which a vacuum of about 4 in. of mercury is maintained. The vapours are condensed. W. N. HOYTE.

Petroleum distillation. W. D. MASON, Assr. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,615,991, 1.2.27. Appl., 11.12.22).—The vapours from the continuously-operated still pass to a tower where they are sprayed with the feed oil; the latter, together with the condensed portion of the vapours, pass to the still. Water is sprayed into the uncondensed vapours in such proportions that it is completely converted into steam. These vapours and steam are then condensed. W. N. HOYTE.

[Fractionating column] for distillation of hydrocarbon oils. M. G. PAULUS and O. C. BREWSTER, Assrs. to STANDARD OIL Co. (U.S.P. 1,615,400, 25.1.27. Appl., 27.2.24).—The column is divided by transverse partitions on which a quantity of liquid is retained; vapours pass up through the partition, and are deflected down through the liquid by means of a cap which rotates; baffles are fitted to prevent vortices forming in the liquid on the partition. W. N. HOYTE.

Oil-cracking still. E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,615,583, 25.1.27. Appl., 28.11.21).—The still consists of a series of pipes exposed to a current of hot flue gases, the oil flowing through counter-current to the gas stream. The tubes nearest the firebox are smooth externally, those farthest away are ribbed. W. N. HOYTE.

Manufacture of highly active charcoal. ALGEM. NORIT MAATSCHAPPIJ (F.P. 608,730, 15.12.25. Conv., 11.5.25).—Residual carbon compounds are removed as completely as possible from charcoal prepared from them, and activation of the charcoal is carried on to such an extent that the product is nearly pure carbon. L. A. COLES.

Production of active charcoal. E. BERL (G. P. 433,524, 15.6.23).—Lignite or coal is extracted with hydrogenated naphthalene derivatives, and, after removal of the solvent, the residue is heated to about 1000°, potassium compounds being added if necessary. L. A. COLES.

Manufacture of lampblack. Decomposition of carbonaceous gases. CANADA CARBIDE Co., LTD., Assees. of S. A. WISDOM (Can. P. 260,226—7, 13.8.23).—(A) Mixtures of acetylene with oxidising gases in quantity insufficient for its complete combustion are heated to

the dissociation temperature of acetylene. (B) Mixtures of carbonaceous gases, of which one is endothermal, are heated to dissociation temperature, so that the heat of dissociation of the endothermal gas causes decomposition of at least a portion of the other constituents of the mixture. L. A. COLES.

Process and apparatus for the manufacture of oil gas. ANC. ETABL. BARBIER, BÉNARD, ET TURENNE (SOC. ANON.) (E.P. 260,215, 26.7.26. Conv., 22.10.25).—See F.P. 605,407; B., 1926, 813.

Cracking liquid hydrocarbons. H. O. SWOBODA and E. M. RICHARDS, Assrs. to H. O. SWOBODA, INC. (U.S.P. 1,616,515, 8.2.27. Appl., 6.7.22).—See G.P. 362,461; B., 1923, 260 A.

Gas burners. FRANKFURTER GAS-GES., and E. SCHUMACHER (E.P. 265,041, 13.4.26).

Furnaces burning pulverised fuel. J. BRASS (E.P. 262,977, 27.1.26).

Apparatus for combustion of liquid fuel. E. BECKER (E.P. 262,948, 19.12.25).

Production of acetylene (F.P. 607,059).—See VII.

Bleaching mineral oils (E.P. 245,745).—See XII.

III.—TAR AND TAR PRODUCTS.

Calcium salts of low-temperature tar phenols. F. R. GREENBAUM (Amer. J. Pharm., 1927, **99**, 10—12).—Calcium phenoxides were obtained in maximum yield by heating a mixture of 450 g. of distilled low-temperature tar phenols, 170 g. of calcium chloride, and 115 g. of calcium hydroxide with 300 c.c. of water, with constant stirring for 6 hrs. under a reflux condenser. The product was filtered off, and washed with water and benzene to remove free phenols; the residue (269 g.) contained 55% of calcium phenoxides and 45% of calcium hydroxide. The product might be of use as an insecticide. L. A. COLES.

PATENTS.

Production of low-temperature tar from bituminous shale. J. FABIAN (Austr. P. 103,714, 10.3.23).—Shale residues are rendered suitable for building purposes, and low-temperature tar is obtained in good yield, by burning away the fixed carbon in the distillation residues, and using the combustion gases, without admixture with steam or other gases, for the distillation of fresh shale. L. A. COLES.

Cracking tar etc. COMP. DES MINES DE VICOIGNE, NOEUX, ET DROCOURT (F.P. 607,279, 13.3.25).—Raw mineral oils, their fractions, tars, or their neutral oil fractions are introduced in a finely-divided state into a melt maintained at the cracking temperature. A. B. MANNING.

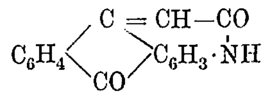
Treatment of light tar-oils, naphtha, or oil distillates, containing unsaturated hydrocarbons such as coumarone and indone. COMP. DES MINES DE VICOIGNE, NOEUX, ET DROCOURT (F.P. 607,672, 21.3.25).—The oils are subjected to a polymerisation process in the presence of oil-soluble metal chlorides, such as titanous chloride, and the purified oil is separated by distillation from resins formed during the process. L. A. COLES.

IV.—DYESTUFFS AND INTERMEDIATES.

Detection and determination of foreign substances in dyes. A. BOHANES (Chem. Listy, 1927, 21, 56—58).—Material insoluble in ether is determined by extraction, and ash by calcination. Soluble salts are separated by dialysis, and determined in the solution. Chlorides are best determined by Votoček's method of titration with mercuric nitrate (cf. B., 1918, 444 A; 1922, 1001 A). S. I. LEVY.

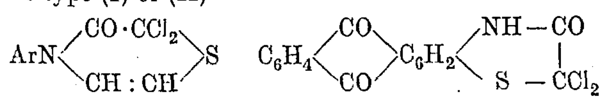
PATENTS.

[Dyes for] dyeing acetyl [cellulose acetate] silk. BRITISH ALIZARINE CO., LTD., W. H. DAWSON, and C. W. SOUTAR (E.P. 263,946, 8.10.25).—Anthrapyridone compounds of the type



prepared by cyclisation of an α-acetamidoanthraquinone, are satisfactory dyes for cellulose acetate silk, being applied in aqueous suspension or colloidal solution. The cyclisation is effected by boiling the acetyl compound with a large excess of dilute caustic soda, or, in some instances, with water alone. *E.g.*, a bright red *dye* is obtained by boiling 300 pts. of 1 : 4-bisacetamidoanthraquinone for about 6 hrs. Reddish-yellow, bright orange, and blue *dyes* are obtained by cyclisation of diacetylamino-, 1 : 5-bisacetamido-, and 1 : 5-diamino-4 : 8-bisacetamidoanthraquinone, respectively. A. J. HALL.

Anthraquinone vat dyes. I. G. FARBENIND. A.-G., Assees. of F. FUNCKE (G.P. 430,901, 28.9.24).—*o*-Diamines are condensed with suitable dichloroketodihydrothiazines of the type (I) or (II)



(I) (II)

to give vat dyes, the diamine or the thiazine, or both, being anthraquinone derivatives; the products may afterwards be alkylated or arylated. 2 : 2-Dichloro-3-keto-7 : 8-phthalylbenzo-2 : 3-dihydro-*p*-thiazine [dichloroketoanthraquinone-2 : 1-dihydrothiazine] condenses in dichlorobenzene at 125—130° with *o*-phenylenediamine to give a brown vat *dye*, m.p. above 300°, with 4 : 5-diamino-*m*-xylene a dark brown vat *dye*, and with 1 : 2-diaminoanthraquinone a yellow vat *dye*. 1-*Amino-2-p-tolylaminoanthraquinone*, from 2-bromo-1-aminoanthraquinone and *p*-toluidine, condenses with the same thiazine in nitrobenzene at 150° to give a greenish-yellow vat *dye*. Dichloroketo-*p*-tolyldihydro-*p*-thiazine (I, Ar = C_7H_7) with 1 : 2-diaminoanthraquinone yields a yellow vat *dye*, and a yellow vat *dye* is also obtained by ethylation with ethyl *p*-toluenesulphonate of the condensation product from dichloroketoanthraquinone-1 : 2-dihydrothiazine and 1 : 2-diaminoanthraquinone. C. HOLLINS.

Anthraquinone vat dyes. I. G. FARBENIND. A.-G., Assees. of K. H. MEYER and H. HOPFF (G.P. 432,579, 29.8.24).—β-Naphthoyl derivatives (containing no substituent hydroxyl group) of aminoanthraquinones containing at least one α-amino-group are prepared by the

action of the acid chloride on the base in nitrobenzene, and are used as vat dyes. β-Naphthoyl chloride with α-aminoanthraquinone gives a greenish-yellow vat dye; with 1 : 5-, 1 : 6-, or 1 : 7-diaminoanthraquinone, yellow; with 1 : 4-diaminoanthraquinone, bluish-red. 2 : 3-Methoxynaphthoyl chloride with 1 : 4-diaminoanthraquinone gives an orange vat dye; 1 : 2-chloronaphthoyl chloride and 1 : 4-diaminoanthraquinone a red. C. Hollins.

Vat dyes of the anthraquinone series. Badische Anilin u. Soda-Fabr. (F.P. 604,347, 7.10.25. Conv., 16.2 and 27.8.25).—Aminoanthraquinones are acylated with *m*-substituted benzoic acids, especially *m*-alkoxybenzoic acids, the dyes obtained being distinguished by good fastness to light. Acylation with *m*-methoxybenzoyl chloride of 1 : 4-diaminoanthraquinone yields a bluish-red dye, of 1 : 5-diaminoanthraquinone a yellow, of 1 : 5-diamino-4-hydroxyanthraquinone a red, of 1 : 5-diamino-4-methoxyanthraquinone an orange, of 4 : 8-diaminoanthrarufin a violet-blue, and of 1-amino-4-methoxyanthraquinone a scarlet. Examples are also given of the use of *m*-toluic, *iso*phthalic, *m*-benzamidobenzoic, 3-methylthiolbenzoic, 5-methoxy*iso*phthalic, and *mm'*-diphenyldicarboxylic acids as acylating agents. A. Davidson.

Manufacture of vat dyes. L. Cassella & Co. G.m.b.H. (F.P. 604,450, 12.10.25).—2-Methylanthraquinone is heated with diamines (other than *p*-diamines) and sulphur. *E.g.*, by heating 2-methylanthraquinone with 1 : 5-naphthylenediamine and sulphur at 240—250° a *dye* is formed which, after extraction with sodium sulphide and purification with sodium hypochlorite, dyes cotton from the vat in reddish-yellow shades. By heating 2-methylanthraquinone with diaminobenzanthrone and sulphur an orange-brown vat *dye* is formed. A. Davidson.

Vat dyes [of the thioindigo series]. H. Wagner, R. Brune, M. Hessenland, E. Hoffa, and F. Müller, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,610,539, 14.12.26. Appl., 3.5.26. Conv., 6.5.25).—2 : 3-Dihalogenotolyl-5-thioglycollic acids, in which position 6 may, or may not, be substituted by CO_2H, CN, or $CO\cdot NH_2$, are converted into the corresponding oxythionaphthens, which are oxidised to the corresponding thioindigo dyes. These compounds give on cotton in the vat shades which are fast to light and kiering. *E.g.*, 6-chloro-*o*-toluidine is converted by the methods described in E.P. 17,417/14 and G.P. 364,822 (B., 1921, 619 a; 1923, 598 a) into 2 : 3-dichloro-6-aminotolyl-5-thioglycollic acid, the amino-group is eliminated, and the product is treated with chlorosulphonic acid, and oxidised to the corresponding 5 : 6 : 5′ : 6′-tetrachloro-4 : 4′-dimethylthioindigo, which dyes cotton in the vat bright, bluish-red shades. The dye is also obtained from 2 : 3-dichloro-6-cyanotolyl-5-thioglycollic acid, which is transformed by treatment with aqueous alkali and sodium sulphide into 5 : 6-dichloro-3-amino-4-methylthionaphthen-2-carboxylic acid; this on heating at 100°, gives 5 : 6-*dichloro-3-oxy-4-methylthionaphthen*, m.p. 157°, which is oxidised by air in alkaline solution in presence of copper. T. S. Wheeler.

Azo dyes. I. G. Farbenind. A.-G., Assees. of H. Krzikalla (G.P. 431,773, 11.12.24).—Sulphonated diazo compounds are coupled with bisacylacetyl derivatives of aromatic diamines to give acid wool dyes fast to washing and milling, or lake dyes, or ice-colours on the fibre. Bisacetoacetyl-*o*-tolidine gives with diazotised sulphanilic acid a greenish-yellow wool dye; with diazotised 6-chloro-*m*-toluidine-4-sulphonic acid a yellow on wool, or as an ice-colour on cotton the dye has good fastness properties. C. Hollins.

Dyes from perylene. Kalle & Co. A.-G., Assees. of M. P. Schmidt and J. Voss (G.P. 432,178, 4.12.23).—Sulphonation of perylene with concentrated sulphuric acid gives at 60° *perylenedisulphonic acid*, a greenish-yellow acid wool dye; at 190° a dark brown vat *dye* for cotton. By the action of sulphuric acid and manganese dioxide at room temperature perylene is converted into a vat *dye*, which gives a yellow-brown on wool. A brown vat dye for wool is obtained by the action of chlorosulphonic acid. Treatment of perylene derivatives with these reagents is also covered. C. Hollins.

Production of 2 : 5-diaminobenzene-1 : 4-dicarboxylic acid (*p*-diaminoterephthalic acid). G. Schroeter (G.P. 433,277, 20.7.24).—Benzene-1 : 2 : 4 : 5-tetracarboxylic acid di-imide (pyromellitic acid di-imide) is shaken with dilute alkaline hypochlorite solution at temperatures below 50° until dissolved; addition of acid reducing agents yields 2 : 5-*diaminobenzene*-1 : 4-*dicarboxylic acid* in quantitative yield as a light yellow, microcrystalline powder (*diethyl* ester, m.p. 168°). E. H. Sharples.

Preparing [3-]nitro[-4-]aminobenzoyl-*o*-benzoic acid and derivatives. R. Adams, J. M. Davidson, and I. Gubelmann, Assrs. to Newport Co. (U.S.P. 1,614,584, 18.1.27. Appl., 8.2.26).—4-Chloro-3-nitrobenzoyl-*o*-benzoic acid on heating with aqueous ammonia at 100° for several hours yields 3-*nitro*-4-*aminobenzoyl*-o-*benzoic* acid, m.p. 224°. In place of ammonia, alkyl- or aryl-amines can be employed. T. S. Wheeler.

Manufacture of water-insoluble azo dyes. Chem. Fabr. Griesheim-Elektron (F.P. 601,687, 5.8.25. Conv., 16.8.24).—Diazo, tetrazo, or diazoazo compounds containing no sulpho- or carboxyl groups are coupled with diarylides of 2-naphthol-3 : 6-dicarboxylic acid. The following diarylides of 2-naphthol-3 : 6-dicarboxylic acid, prepared in the usual way, are described:—*dianilide*, m.p. 282°; *di-o-toluidide*, m.p. 255°; *di*-m-*chloroanilide*, m.p. 243—244°; *di-o-anisidide*, m.p. 150—152°; *di-α-naphthylamide*, m.p. 218—220°. A bluish-red lake is obtained by coupling diazotised *p*-nitro-*o*-aminotoluene with the dianilide of 2-naphthol-3 : 6 : dicarboxylic acid. Cotton padded with the dianilide and developed with diazotised 4-nitro-2-aminoanisole gives fast bluish-red shades. A. Davidson.

Non-alkaline solutions or pastes of sulphide dyes. I. G. Farbenind. A.-G., Assees. of H. Kesseler and E. Döring (G.P. 432,112, 15.3.24).—Solutions or pastes of sulphide dyes suitable for dyeing animal fibres and acetate silks or for printing on account of their non-alkaline reaction, are made by treatment with magnesium hydroxide and a reducing agent, such as sodium hyposulphite. C. Hollins.

Azo dyes. I. G. Farbenind. A.-G., Assees. of L. Laska and A. Zitscher (G.P. 430,579, 21.9.24).—Azo dyes, suitable especially as ice-colours, are made by coupling diazotised aminodiphenyls (2- and 4-aminodiphenyl, aminoditolyls, and their chloro- and dichloro-derivatives) with 2 : 3-hydroxynaphthoic arylamides. 4 : 4'-*Dichloro*-3 (?)-*aminodiphenyl*, m.p. 95—96°, and 4 : 4'-*dichloro*-6-*amino*-3 : 3'-*dimethyldiphenyl*, m.p. 88°, are prepared by reduction of nitrated 4 : 4'-dichlorodiphenyl and 4 : 4'-dichloro-3 : 3'-dimethyldiphenyl, respectively; 4-*amino*-4'-*hydroxydiphenyl*, m.p. 273°, from benzidine by diazotising one amino-group and boiling the diazo-solution. 2 : 3-Hydroxynaphthoic *m*-chloroanilide, coupled on the fibre with diazotised 4-chloro-4'-aminodiphenyl, gives a garnet shade. All the dyeings are fast to bowking. C. Hollins.

Brown dyes for wool and leather. I. G. Farbenind. A.-G., Assees. of R. Schmidlin (G.P. 431,776, 29.5.24. Addn. to 414,390).—A diaminodiphenyl or diaminodiphenylmethane derivative is condensed first with 1 mol. proportion of a halogenonitroaryl-sulphonic or -carboxylic acid, and then with a quinone of the benzene series. Thus, from equimolecular proportions of benzidine, 1-chloro-4-nitrobenzene-6-sulphonic acid, and toluquinone a tobacco-brown *dye* for leather is obtained. C. Hollins.

Fuel-testing composition (U.S.P. 1,615,143).—See II.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Degradation of the cellulose and lignin of wood, two distinct processes of decomposition by mycelium present in wood. R. Falck and W. Haag (Ber., 1927, **60** [*B*], 225—232).—The decay of wood is due to corrosion generally caused by parasitic agents and destruction brought about by species of *merulius*, *coniophora*, *poria*, *lenzites*, etc. During corrosion the lignin is first affected, followed by the cellulose, a considerable proportion of which remains after the lignin has disappeared completely. During destruction, the difference is more pronounced, since the cellulose disappears completely, whereas the lignin is unaffected. These differences also find expression in the carbon content of the products at various stages. In both series the pentosan content decreases uniformly. The copper numbers in the series are quite distinct, and show that the enzyme does not cause accumulation of oxycellulose or similar degraded products. The increased copper number of merulius cellulose proves that insoluble compounds with reducing properties are initially produced; acidic substances are also formed, quantitatively, similar to those obtained by the action of *Aspergillus niger* on starch. The aqueous extracts of corroded wood (at any rate until decomposition is very profound) resemble those of sound wood, whereas those from "destroyed" wood are dark brown, acidic, and reducing in the early stages of the process. During corrosion there is a decrease in the proportion of substance soluble in alcohol–benzene, whereas the reverse is the case during destruction. The possibility of isolating lignin from wood by the aid of *merulius* species under milder conditions than those employed by Willstätter and Zechmeister is indicated. H. Wren.

Gelatinisation of lignocellulose. III. Viscose reaction. A. W. Schorger (Ind. Eng. Chem., 1927, **19**, 226—227; cf. B., 1923, 923 A; 1924, 250).—Owing partially to filtration difficulties, attempts to obtain lignin in an unchanged state from wood by means of the viscose reaction proved unsuccessful. A large part of the lignin passed into solution with the carbohydrate, and of the 82% of aspen wood and 70% of white pine dissolved, a considerable proportion was not precipitated on acidification. The lignin of white pine was more soluble than that of aspen. Cellulose is more easily removed from hydrolysed than from the original wood. C. O. Harvey.

Chemistry of the sulphite process. V. Digestion of spruce wood with sodium bisulphite. E. Hägglund (Papier-Fabr., 1926, **24**, 775—780).—Digestion with sodium bisulphite takes place with somewhat more difficulty than with the calcium liquor. A 12% solution of sodium bisulphite at 130° or at 140° gives practical products. There is exceptional liability to "black cooks" with concentrated liquors, *e.g.*, with 18% solution of sodium bisulphite at 140°. This tendency is attributed to the low hydrogen-ion concentration of the sodium bisulphite liquor, since high initial hydrogen-ion concentration protects against this decomposition. On the other hand, the yield of cellulose is extremely high. The first stage of the process consisting in the combination of the bisulphite with the lignin–carbohydrate constituent takes place very rapidly at a low temperature. The second stage, during which this solid lignosulphonic acid is hydrolysed, proceeds slowly under normal conditions, so long as decomposition of the bisulphite liquor and formation of sulphuric acid are negligible. At this stage the ratio of carbohydrates to lignin in the liquor remains normal and constant. In cooks carried out without blackening, the cellulose is characterised by a high content of insoluble lignosulphonic acid, equivalent to 3·5—7·0% of lignin, or 13—25% of the original lignin of the wood. The ratio of reducing sugars after inversion, to direct reducing sugars in the liquor, remains at about 4·0. Notable destruction of pentosans occurs even in the early stages of digestion, but the lignin of wood contains 1·24% of substance yielding furfuraldehyde expressed as pentosan which is not a pentosan. The pulps from the sodium bisulphite digestion are easily resolved into fibres, but on account of their high content of insoluble lignosulphonic acid, they belong rather to the class of "half celluloses," and yield papers of very high strength and folding qualities. J. F. Briggs.

Determination of water in sulphite pulp. E. Schlumberger (Papier-Fabr., 1926, **24**, 783—785).—In the determination of moisture in cellulose by the distillation method, tetrachloroethane is used instead of an inflammable hydrocarbon. The apparatus consists of a boiling flask of 700 c.c. capacity, with ground-in stopper carrying a wide, vertical, distillation column, bent round into a vertical condenser. The distillate is delivered into a U-shaped receiver consisting of two bulbs acting as a Florentine separator and extending at its inlet into a vertical measuring tube or burette 20 cm. long and 3 mm. wide, graduated in 0·02 c.c. The cellulose is boiled with tetrachloroethane, and distillation

is continued until the drops of distillate previously cloudy again come over clear; this takes about ½ hr. at a distillation rate of 80—100 c.c. in 10 min. The volume of water is read off in the measuring tube with a maximum error of $\pm$ 0·17%. J. F. BRIGGS.

PATENTS.

Production of fibres capable of being spun. OBERRHEINISCHE HANDELS GES.M.B.H., and L. UBBELOHDE (E.P. 264,233 and 264,428, 14.10.25. [A] Addn. to 254,357; B., 1926, 782).—(A) The roughening and curling of smooth fibres in order to increase their spinning qualities are produced by precipitating inorganic or organic roughening agents on or within the fibres by chemical decomposition or physical means in the wet, semi-dry, or dry state as in mordanting or waterproofing, or by causing the adherence of an organic colloid. (B) The fibres are rendered more sensitive to the roughening material by treatment with alkalis, acids, salts, solvents, or swelling agents at some or all stages of the manufacturing process. The fibres are mixed with a suspension of the roughening agent (infusorial earth, kaolin, silica gel, sand, lime, etc.) in the sensitising solution, agitated, and dried after removal of part of the liquid. Agitation alone after sensitising also produces roughening. B. P. RIDGE.

Production of pattern effects on fabrics. TOOTAL BROADHURST LEE CO., LTD., and R. P. FOULDS (E.P. 264,559, 9.10.25).—The material (artificial silk, cotton, or mixtures of both) is treated in selected areas with a concentrated solution of antimony or bismuth trichloride, and subsequently washed with water, more concentrated solutions being required for cotton than for artificial silks. The chloride is dissolved in glycerin, lactic acid, or any other medium from which the oxide is not thrown out until the fabric is treated with water, and a thickening agent suitable for use in an acid medium, *e.g.*, china clay and gum, is added if required. Antimony trichloride with feculose and lactic acid gives the best results. B. P. RIDGE.

Treatment [sizing] of yarns or threads. BRITISH CELANESE, LTD., C. W. ADDY, J. BILLING, and H. HALKYARD (E.P. 264,382, 4.6.26).—Yarns in packages or on bobbins etc. are impregnated in an airtight chamber with a solution of a sizing or dressing agent in a volatile solvent, penetration being assisted by evacuation or pressure or both used alternately. After removal from the solution they are unwound while still wet, dried by evaporation of the solvent, and re-wound on to bobbins or hanking frames. Suitable sizing compositions are described in E.P. 244,947 and 244,979 (B., 1926, 189). B. P. RIDGE.

Treatment of wool for the purpose of diminishing its affinity for acid and neutral-dyeing wool dyes. I. G. FARBENIND. A.-G., Assees. of A. GÜNTHER, W. SCHLEGEL, and A. THAUSS (G.P. 432,111, 20.6.23).—Clear two-colour effects are obtained on half-wool materials by pre-treating the wool with sulphurised phenol products with or without the addition of tin salts (cf. G.P. 409,782; B., 1925, 627), so that it has less affinity for acid and neutral-dyeing wool dyes, or by the addition of sulphurised phenols to the dye bath. A. J. HALL.

Production of wool-like effects on cotton fabrics. H. I. HUEY, Assr. to SAYLES FINISHING PLANTS, INC. (U.S.P. 1,616,749, 8.2.27. Appl., 5.5.22. Renewed 30.9.24).—The unmercerised material is first treated with sulphuric acid of *d* 1·547—1·580, washed, dried, and afterwards treated with sodium hydroxide solution of *d* 1·116—1·180 at 30—35°, with subsequent washing. B. P. RIDGE.

Substitutes for catgut and similar animal products. DUNLOP RUBBER CO., LTD., and R. TRUESDALE (E.P. 264,640, 11.12.25).—Threads formed from any textile fibres are impregnated with a viscous cellulose solution, such as cellulose xanthate, drawn through die boxes, and twisted together, a further coating of solution being given if desired. The solution is coagulated and the cords are dried by passing through a heated chamber. Composite cords are produced by twisting the impregnated threads round a suitable core, *e.g.*, catgut or wire, and flexibility is obtained by the addition of a softener such as glycerin to the cellulose solution. B. P. RIDGE.

Manufacture of textile fabrics [resembling crêpe]. H. DREYFUS (U.S.P. 1,614,832, 18.1.27. Appl., 9.4.24. Conv., 4.9.23. Cf. E.P. 224,642).—The process of E.P. 226,256 (B., 1925, 201) is applied to fabrics containing reconstituted cellulose, *e.g.*, denitrated nitrocellulose or cellulose recovered from cuprammonium solutions. T. S. WHEELER.

Washing and cleaning fabrics. H. SPINDLER (U.S.P. 1,615,146, 18.1.27. Appl., 1.6.23. Conv., 1.6.22).—Materials can be satisfactorily washed in hard water, using a soap containing a finely-divided oxide or hydroxide of magnesium, calcium, barium, aluminium, zinc, or similar weakly-basic, water-insoluble, metal compound, capable of precipitating calcium and magnesium compounds from the water, and thus softening it in the washing process. The soap can be prepared by adding a soluble salt of the metal to the fatty acids before saponification. T. S. WHEELER.

Process and apparatus for bleaching cellulose pulp and similar fibrous materials. C. B. THORNE (E.P. 264,596, 21.10.25).—Concentrated pulp is treated uniformly with a bleaching agent and water in a mixer, and then, while still comparatively stiff, is conveyed continuously downwards through a series of aerating towers, through which air or gas is passed from a slowly-rotating hollow shaft carrying a perforated distributing arm, agitation of the pulp being avoided as much as possible. Removal from each tower is facilitated by the use of a jet of water, the material being again concentrated before entering the next tower. Bleaching of highly-concentrated pulp may be combined with the treatment of diluted material. B. P. RIDGE.

Cellulose ester composition. J. G. DAVIDSON, Assr. to CARBIDE AND CARBON CHEMICALS CORP. (U.S.P. 1,617,237, 8.2.27. Appl., 10.5.26).—In a pyroxylin plastic, dibenzyl is used as a plasticiser. Nitrocellulose (100 pts.), esters of the higher alcohols (10—20 pts.), and dibenzyl (10—30 pts.) are mixed with sufficient acetone to give the desired plasticity. B. P. RIDGE.

[Non-inflammable] cellulose composition. W. G. LINDSAY, Assr. to CELLULOID CO. (U.S.P. 1,616,910,

8.2.27. Appl., 25.4.22).—Lithium phosphate containing water of crystallisation is mixed with a homogeneous, non-fibrous, cellulosic derivative in such proportion as to render the composition non-inflammable.

B. P. Ridge.

Viscose-treating apparatus. S. A. Neidich (U.S.P. 1,616,918, 8.2.27. Appl., 10.5.26).—A circular series of trays, capable of rotation in a horizontal plane about a common axis, is arranged above a corresponding series of tanks, means being provided for the intermittent discharge of liquid from the trays into the tanks and *vice versa*, discharge of the liquid and rotation of the trays taking place alternately. B. P. Ridge.

Utilisation of black liquor. E. Hägglund (Can. P. 260,376, 20.7.25).—After subjection to heat and pressure, the liquor is mixed with fresh sodium carbonate solution, and used for treating fibrous material. L. A. Coles.

Cooling and utilisation of the heat content of relief gas from sulphite digesters. G. A. Richter, Assr. to Brown Co. (U.S.P. 1,616,703, 8.2.27. Appl., 28.4.25).—The hot relief gas from the digesters is washed with an opposing flow of cold water acidified with sulphur dioxide from an independent source, thereby causing cooling, condensation of steam, and liberation of sulphur dioxide from the water, and yielding a cool gas rich in free sulphur dioxide. B. P. Ridge.

Drying of fabrics in the open width. S. F. Barclay, and Mather & Platt, Ltd. (E.P. 265,026, 18.3.26).

Cellulose ester compositions (U.S.P. 1,611,169 and 1,612,669).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Recent developments in the dyeing of Celanese [cellulose acetate silk] mixtures. C. G. Candlish (J. Soc. Dyers and Col., 1927, **43**, 37—42).—Grey Celanese contains little impurity beyond traces of lubricating oil, and requires only a mild scour before dyeing. Bleaching is carried out by the usual methods, or may be effected simultaneously with scouring at 60°. Celanese materials are transparent to ultra-violet light, even after being de-lustred by treatment with boiling water. When Celanese is immersed in boiling water it loses its lustre; use is made of this fact to produce artificial silk materials having a subdued lustre. De-lustreing is assisted by the presence of soap, Turkey-red oil, or other substances capable of reducing the surface tension of the boiling liquor. The use of Celanese in worsted goods is limited because of the loss of lustre during such processes as crabbing and steaming, but the presence of inorganic salts such as magnesium chloride, ammonium sulphate, or sodium chloride assists preservation of the lustre (cf. E.P. 246,879; B., 1926, 317). Vat dyes may be applied to cotton materials containing Celanese without hydrolysis of the latter, provided that phenol is added to the dye bath sufficient to combine with excess of caustic alkali. A. J. Hall.

Patents.

Dyeing or colouring of yarns and fabrics or other materials. British Celanese, Ltd., and G. H. Ellis (E.P. 263,473, 7.9.25).—In dyeing textile materials, containing animal or other fibres deleteriously affected by caustic alkalis, with vat or sulphur dyes, the caustic alkali usually employed in the dye liquor is replaced wholly or partly by the alkali metal or ammonium salts of hydroxy-, polyhydroxy-, or simple-ring-substituted derivatives of polyhydroxy- or hydroxy-compounds of the homocyclic or heterocyclic organic series, *e.g.*, sodium phenoxide, potassium tolyloxide, sodium quinoxide, sodium β-naphthoxide, and sodium *p*-chlorophenoxide (cf., E.P. 262,506; B., 1927, 105).

A. J. Hall.

Fixing basic dyes on [textile] fibres and other substrata. I. G. Farbenind. A.-G., Assees. of H. Bernard (G.P. 432,113, 30.8.24).—Oxidation products (of higher mol. wt.) of phenols or their homologues or substituted derivatives are satisfactory substitutes for tannic acid in fixing basic dyes (cf. G.P. 406,675; B., 1925, 413). *E.g.*, fabric (unmordanted) is impregnated with a basic dye and the latter fixed by passage through a solution of an oxidised phenol, or colour lakes are formed by bringing together solutions of basic dyes and of an oxidised phenol in the presence of a suitable substratum. A. J. Hall.

Dyeing animal fibres in acid baths. L. Cassella & Co., G.m.b.H., Assees. of R. Haynn (G.P. 433,145, 28.9.24).—Aldehydes, *e.g.*, formaldehyde, or substances which liberate aldehydes, are added to acid dye-baths used for animal fibres for the purpose of preserving the structural qualities of the fibres. A. J. Hall.

Cellulose derivatives. I. G. Farbenind A.-G., Assees of F. Günther (G.P. 433,147, 29.6.24).—Cellulose or a related substance is treated with isatoic anhydride or compounds containing the characteristic group of isatoic anhydride, and gives a material which may be diazotised and coupled with the usual components. An isatoic anhydride which already contains an azo grouping may be used. Cotton treated with isatoic anhydride becomes orange when diazotised and coupled with β-naphthol. Treatment of cotton with *p*-sulphobenzeneazoisatoic anhydride (prepared by phosgenation of *p*-sulphobenzeneazoanthranilic acid), followed by diazotisation and coupling with β-naphthol, gives a red. A yellow results when cotton is treated with the isatoic anhydride obtained by phosgenation of the azo dye from 2-chloroaniline-5-sulphonic acid diazotised and coupled with anthranilic acid; the cotton so treated may further be diazotised and coupled with the usual components. C. Hollins.

Dyeing cellulose esters. I. G. Farbenind. A.-G., Assees. of F. Günther (G.P. 432,110, 26.4.22).—Cellulose esters are dyed by means of formaldehyde-bisulphite compounds of azo dyes containing a primary amino-group. If the dye contains a diazotisable amino-group, the dyed material may be further diazotised and coupled with suitable components. A. J. Hall.

Dyeing of cellulose esters or ethers. I. G. Farbenind. A.-G., Assees. of W. Duisberg, W. Hentrich, C. Weinand, and L. Zeh (G.P. 433,236, 27.6.24).—1-Aminoanthraquinone-2-sulphonic acids, excepting those containing in the 4-position a free or substituted amino- or hydroxyl group, are claimed as dyes for artificial silks made from cellulose esters or ethers; *e.g.*, 1-amino-

anthraquinone-2-sulphonic acid (orange), its 4-bromo- (yellowish-red), 4-tolylthiol- (reddish-violet), and 5-*p*-tolylamino- (bordeaux) derivatives. C. HOLLINS.

Dyeing of leather. V. CASABURI (Swiss P. 114,271, 11.10.24).—Leather is mordanted with organic complex metallic salts of aromatic sulphonic acids containing amino- and hydroxyl groups in the nucleus and then dyed, or the dyeing and mordanting can be carried out simultaneously. The mordant is prepared, *e.g.*, by mixing 10 pts. of 1-amino-8-naphthol-3 : 6-disulphonic acid with 2·26 pts. of chromium hydroxide, 3 pts. of anhydrous sodium carbonate, 4 pts. of sodium acetate, and 6 pts. of potassium hydrogen sulphate. Chrome leather which has been treated with a solution of this mordant can be dyed with basic and substantive dyes. Vegetable mordanted leather and glove leather can also, after mordanting, be dyed with substantive dyes. In dyeing with substantive dyes, the dye and mordant solutions can be used together. In place of chromium hydroxide, the hydroxides of iron, aluminium, copper, etc. can be used. A. DAVIDSON.

Production of multicoloured and black discharges on leather and artificial leather. I. G. FARBENIND. A.-G., Assees. of R. KLEIN (G.P. 433,154, 28.4.25).—Dressed leather is dyed with suitable dyes, then dried, printed with a paste containing a discharging agent such as formaldehyde-sulphoxylate or sodium hyposulphite compounds (zinc may be present) and suitable natural or synthetic dyes, then dried and steamed for 1½ min. A. J. HALL.

Dyeing, printing, and stencilling of cellulose acetate or products made therewith. BRITISH CELANESE, LTD., and G. H. ELLIS (E.P. 263,260, 23.10.25).—Cellulose acetate silk materials are dyed with acidyl derivatives of aminoanthraquinones or their methylamino-, hydroxy-, chloro-, or methyl derivatives, applied in a solubilised form (cf. E.P. 219,349; B., 1924, 906), or by vatting methods such as are ordinarily used for vat dyes or by special vatting methods (cf. E.P. 262,506; B., 1927, 105) in which minimum quantities of caustic alkali are used. Suitable acylamino-anthraquinone dyes include 1-acetamidoanthraquinone (lemon-yellow), 1-propionamidoanthraquinone (greenish-yellow), 1-acetamido-4-methoxyanthraquinone (golden-yellow), 1-acetamido-4-hydroxyanthraquinone (orange), 1-acetmethylamido-4-methoxyanthraquinone (orange), 1 : 4-bisacetamidoanthraquinone (orange), 1 : 4-bispropionmethylamidoanthraquinone (bright red), and 1-acetamido-4-methylaminoanthraquinone (reddish-violet). These acidyl dyes have a smaller affinity for vegetable and animal fibres. A. J. HALL.

Producing fast printings [on textile materials]. A. G. BLOXAM. From I. G. FARBENIND. A.-G. (E.P. 262,987, 11.2.26).—Coloured effects particularly fast to washing are obtained by printing textile materials with a mixture containing an alkali salt of a suitable azo compound and the salt of an aromatic sulphonitrosoaminic acid containing one or more of the grouping $\cdot N(NO)\cdot SO_3M$ (M = a metal), and then developing (with simultaneous elimination of the sulpho-group) the shade by after-treatment with acid vapours or an aqueous solution of a weak acid such as acetic or formic acid. Alternatively, the sulphonitrosoaminic acid may be formed *in situ* by substituting for it in the printing paste a mixture of sodium nitrite and an alkali salt of an aromatic sulphamic acid. Sulphonitrosoaminic acids derived from amino- or diamino-compounds of diaryls or of the aromatic series, which contain azo, azoxy, carbamide, thiazole, iminazole, 4-azoimido-, or similar groups, are suitable, and particularly suitable are the salts of sulphonitrosoaminic acids of aminoazo compounds having the general formula, $R\cdot N:N\cdot R'\cdot N(NO)\cdot SO_3M$ (R and R' = aromatic residues). Suitable azo compounds include naphthols and their derivatives, *e.g.*, the arylides of 2 : 3-hydroxynaphthoic acid, derivatives of α-naphthol-4-carboxylic acid, and, further, such substances as contain a reactive methylene group, *e.g.*, pyrazolone derivatives or derivatives of αβ-keto-aldehydes. A. J. HALL.

Printing fast shades [on textile materials]. I. G. FARBENIND. A.-G., Assees. of A. ZITSCHER and F. MURIS (G.P. 433,276, 25.12.24).—Fabric is printed with a mixture containing an alkali salt of a suitable azo component and a sulphonitrosoamine derivative of an aminoazo compound having the general formula $R\cdot N:N\cdot R'\cdot N(NO)\cdot SO_3M$ (R and R' are aromatic residues), then steamed, and successively passed through acid and alkaline liquors for the development of the pigment dye within the fibre. Alternatively, the sulphonitrosoamine compound is replaced by a mixture containing an alkali salt of a sulphoaminoazo compound and sodium nitrite. *E.g.*, a deep black shade is obtained by printing cotton fabric with a paste containing the sodium salt of *o*-phenetoleazonaphthylsulphonitrosoamine, 2 : 3-hydroxynaphthoyl-*m*-nitroaniline, Turkey-red oil, caustic soda, and starch thickening, then steaming and passing successively through dilute hydrochloric acid and caustic soda, afterwards washing and soaping. Such printing mixtures may be used simultaneously with other well-known nitrosoamine printing pastes. A. J. HALL.

Printing processes [for textile materials]. I. G. FARBENIND. A.-G., Assees. of K. H. MEYER, H. KRZIKALLA, and A. SCHNEEVOIGT (G.P. 433,153, 18.11.24).—Methylolformamide is a good solvent for basic and other dyes, and is miscible with the thickening agents usually employed in printing pastes. Coloured effects obtainable in deep shades, and having a fastness to washing superior to that of similar effects obtained with the assistance of tannic acid, are produced by printing fabric with a paste containing methylolformamide, basic or other dyes, and one or more aromatic hydroxy-compounds, and then steaming or drying on drying cylinders, after-treatment in a fixing bath of tartar emetic being unnecessary. A. J. HALL.

Dyeing acetyl cellulose or fabrics containing the same and new products for use therein. J. BADDILEY, A. SHEPHERDSON, H. SWANN, J. HILL, and L. G. LAWRIE, Assrs. to BRITISH DYESTUFFS CORP., LTD. (U.S.P. 1,616,103, 1.2.27. Appl., 19.10.25. Conv., 7.1.25).—See E.P. 246,984; B., 1926, 317.

Dyeing cellulose acetate (E.P. 263,946).—See IV.

Solutions or pastes of sulphide dyes (G.P. 432,112).—See IV.

Bleaching cellulose pulp (E.P. 264,596).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Catalytic oxidation of ammonia. III. L. Andrussov (Z. angew. Chem., 1927, **40**, 166—174; cf. A., 1926, 582; B., 1926, 318).—The behaviour of mixtures of ammonia and oxygen at a platinum contact has been examined at temperatures up to 1300°, velocities of 570 cm./sec., and periods of contact as short as millionths of a second. At and above 1200° the yield of nitric oxide may exceed 90% if three successive contacts are used. The course of the change is represented by the equations $NH_3 + O_2 = NH_3O_2 = HNO + H_2O$; $HNO + O_2 \rightarrow NO + H_2O$; $HNO + NH_3 = NH_3HNO = N_2H_2 + H_2O$ and $N_2H_2 + O_2 \rightarrow N_2 + H_2O$. The most important factor influencing the change is the ratio, $O_2 : NH_3$. If this falls below 0·75, nitrogen is almost the sole product under all conditions examined. Only if the ratio exceeds 1·25 is nitric oxide almost quantitatively produced. Between these ratios there is a proportional division of the yield between nitric oxide and ammonia. Particularly when the velocity of the gas current is low, complications ensue owing to the diffusion backwards of the products of the reaction and formation of nitrogen from ammonia and nitroxyl (*v.s.*). H. Wren.

Theory of the catalytic combustion of ammonia. M. Bodenstein (Z. angew. Chem., 1927, **40**, 174—177; cf. Andrussov, preceding abstract).—It is assumed that the primary change is expressed by the equation, $NH_3 + O_2 = HNO + H_2O$, and that the nitroxyl is converted either into nitric acid (which ultimately yields nitric oxide, water, and oxygen), or, by ammonia, into nitrogen, water, and hydrogen. In the absence of sufficient ammonia or oxygen nitroxyl may decompose into (*a*) nitrous oxide and water, (*b*) nitrogen, oxygen, and water, or (*c*) hydrogen and nitric oxide. H. Wren.

Schlagdenhaufen's reaction [for magnesium]. A. Hamy (Ann. Falsif., 1927, **20**, 19—20).—The reaction of Schlagdenhaufen for detection of magnesium occurs under the most favourable conditions in the presence of excess of sodium or potassium, of iodine, and of potassium iodide. 50 c.c. of the sample to be tested are mixed with 6—7 c.c. of *N*-iodine solution dissolved in 20% potassium iodide solution, then 5 c.c. of *N*-potassium hydroxide solution are added and shaken violently for 1 min. The red-brown precipitate formed in the presence of magnesium is filtered through asbestos, and can be decolorised by alcohol. D. G. Hewer.

Thermal and material efficiency of modern calcium carbide and ferrosilicon [electric] furnaces. E. Schlumberger (Z. angew. Chem., 1927, **40**, 141—146).—The data given are derived from a modern 4500-kw. unit in Germany, and represent average good working. In the manufacture of calcium carbide, 78% of the calcium used is present in the finished pure carbide, 10% is found as calcium oxide, and 6% lost as dust and vapour. Of 100 pts. of carbon used together with 7 pts. of electrode carbon consumed, 51 pts. are recovered as carbide or graphite, 25 pts. as carbon monoxide, and 31 pts. lost. The loss is increased by increasing ash content in the coke. Of the power used, 64% is employed in the reaction and 3% in evaporation; 8% represents electrical transformation losses, and 25% radiation and convection losses of heat. In the manufacture of ferrosilicon (45% Si), 98% of the iron is recovered and 88% of the silicon. Of the carbon used in the form of anthracite, wood, charcoal, and coke, 67% is usefully employed in the reduction of silica, 5% being lost in the reduction of ferric oxide in the ash, and the remainder burnt or lost in the form of vapour. The energy efficiency is 63%, the losses being somewhat similar to those in the carbide process. C. Irwin.

Determination of small quantities of hydrogen in nitrogen as hydrogen chloride. G. Heyne (Z. anal. Chem., 1927, **70**, 179—183).—A combustion tube is packed with a column of anhydrous nickel chloride, 20 cm. long; the tube is heated at 600° for several hours while a slow current of anhydrous hydrogen chloride is passed through it, and finally this is displaced with a current of pure nitrogen, so as to effect the complete removal of any traces of water in the nickel chloride. The gas to be tested for hydrogen is then passed slowly through the tube heated at 600°, thence through an absorption tube containing dilute sodium hydroxide solution to collect the hydrogen chloride formed by the reduction of the nickel chloride by the hydrogen in the gas sample. The chloride is determined volumetrically by Volhard's method. Less than 0·001% of hydrogen in nitrogen may be accurately determined in this way. A. R. Powell.

Patents.

Making sulphuric acid. W. F. Lamoreaux (U.S.P. 1,617,212, 8.2.27. Appl., 13.6.25.)—Sulphur dioxide gas brought into contact with a regulated supply of nitrated acid completely denitrates the sulphuric acid produced, the latter being then brought into contact with a current of gas to remove the residual sulphur dioxide, after which the purified acid is collected. H. Royal-Dawson.

Evolution of hydrocyanic acid from cyanides. H. Lehrecke, Assr. to Roessler & Hasslacher Chem. Co. (U.S.P. 1,615,637, 25.1.27. Appl., 21.11.25. Conv., 27.11.24).—A mixture of sodium cyanide, anhydrous magnesium sulphate, magnesium hypochlorite, and potassium bisulphate is treated with water. H. Royal-Dawson.

Making arsenic acid. W. C. Piver (U.S.P. 1,615,193, 18.1.27. Appl., 21.2.23).—An arsenic ore is heated in a current of air, and the volatilised arsenious oxide is passed with air into a tower containing nitric acid and water vapour. Arsenic acid is produced, and the nitrogen oxides formed are reoxidised by the air present. T. S. Wheeler.

Manufacture of phosphoric acid. I. G. Farbenind. A.-G. (F.P. 609,100, 11.1.26. Conv., 17.1.25).—In the manufacture of phosphoric acid by the combustion of phosphorus or gas mixtures containing it, phosphoric acid solution trickles down the walls of the combustion chamber. L. A. Coles.

Manufacture of effervescent alkali compounds. W. F. Little, Assr. to Upjohn Co. (U.S.P. 1,616,587, 8.2.27. Appl., 25.8.26).—Sodium carbonate (5 pts.) and calcium lactate (1 pt.) are mixed with powdered tartaric and citric acids and sufficient additional sodium bicar-

bonate to react with the acids, the mixture being sprayed with 90% alcohol to prevent dust rising from the powder. The mass is then passed to a fusion drier in which the temperature and moisture are controlled to give 93° on the dry bulb and 54° on the wet bulb. After fusion, the mass is broken into granules and dried at 75°.

H. ROYAL-DAWSON.

Manufacture of ammonia. SOC. D'ÉTUDES MINIÈRES & INDUSTRIELLES (E.P. 253,540, 31.12.25. Conv., 8.1.25).—A hot mixture of nitrogen and hydrogen is passed over iron, nickel, cobalt, tungsten, or mixtures of these metals, with the addition of 40% of lithium nitride or amide (cf. B., 1926, 1013), and 5—10% of alumina, magnesia, or lime. Ammonia is formed at 550—600° and under atmospheric pressure. W. G. CAREY.

Production of ammonia and acetylene. L. TOCCO and M. LANDI (F.P. 607,059, 18.11.25. Conv., 17.7.25).—Barium oxide is used instead of the carbonate in Margueritte and Sourdeval's process for the production of barium cyanide, and the cyanide is converted into barium oxide, acetylene mixed with small quantities of other hydrocarbons, and ammonia, by treatment with hydrogen and steam in one or more revolving furnaces.

L. A. COLES.

Manufacture of sodium monoxide. ROESSLER & HASSLACHER CHEMICAL CO. (E.P. 264,724, 7.6.26. Conv., 29.1.26. Addn. to 253,520 ; B., 1927, 187).—Sodium is mixed with finely-divided sodium monoxide in such proportion as not to produce a pasty mass, the mixture containing not over 10% of uncombined sodium, and the mass is oxidised with atmospheric oxygen, the air being regulated so that the oxygen concentration in the reaction vessel is well below 10%. Heating is necessary only to start the reaction, after which the reaction temperature is prevented from rising above 250° by restricting the supply of air. W. G. CAREY.

Dehydration of fused mixtures of alkaline-earth chlorides and magnesium chloride. I. G. FARBENIND. A.-G., Assees. of P. SIEDLER and W. MOSCHEL (G.P. 433,666, 17.2.25).—Granulated calcium carbide is added to the fused material. L. A. COLES.

Preparation of alkali cyanide solutions. K. ANDRICH, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,615,208, 25.1.27. Appl., 22.12.25. Conv., 23.12.24).—Hydrocyanic acid is caused to react with an aqueous mixture of alkali sulphate and calcium hydroxide at about 15°. H. ROYAL-DAWSON.

Manufacture of bisulphites. J. B. BEVERIDGE (Can. P. 259,884, 9.4.25).—Solutions containing sodium and magnesium bisulphites are obtained by treating mixtures of lime and magnesia with sulphur dioxide in the presence of water, adding a quantity of sodium sulphate equivalent to the calcium, and filtering off the precipitated calcium sulphate. L. A. COLES.

Production of metal [lead] phosphate. H. BLUMENBERG, JUN., Assr. to STOCKHOLDERS SYNDICATE (U.S.P. 1,617,098, 8.2.27. Appl., 8.5.25).—A mixture of litharge and ammonium dihydrogen phosphate is heated to dispel the ammonia and water formed. H. ROYAL-DAWSON.

Production of bismuth silicate. J. ZELTNER (G.P. 433,526, 16.9.24).—Aqueous solutions containing bismuth salts are treated with soluble alkali silicates. L. A. COLES.

Conversion of material other than glue into small particles. A.-G. FÜR CHEM. PROD. VORM. H. SCHEIDEMANDEL, D. SAKOM, and P. ASKENASY (G.P. 434,278, 14.5.24).—The material, or a concentrated solution of it, is allowed to fall successively through two immiscible liquids in which it is not soluble. The lower liquid layer, which serves to remove the upper liquid from the particles, may consist of a solution of the material under treatment of such concentration that the material does not dissolve. For example, fused sodium sulphide, or a hot saturated solution of it, is dropped into dichloroethylene containing sufficient tetralin to bring it to *d* 1·150, and the solidified particles sink into saturated sodium sulphide solution (*d* 1·175), whence they are removed. L. A. COLES.

Production of highly-concentrated active hydrogen.—A. THIEL and G. STAMPE (G.P. 433,520, 28.11.25).—A wall of a reaction vessel is constructed of a sheet of metal penetrable by hydrogen, the outer side of the wall serving as the cathode for the electrolysis of water. Tubes having the outer surface constructed of metal penetrable by hydrogen and the inner surface serving as cathode for the electrolysis of water, are used as unpolarisable hydrogen electrodes. L. A. COLES.

Active-chlorine preparation. E. B. PUTT (U.S.P. 1,612,742, 28.12.26. Appl., 22.3.26).—Cineole is employed to mask the odour of preparations containing active chlorine ; *e.g.*, 0·5% is added to solutions of sodium hypochlorite. T. S. WHEELER.

Recovery of chlorine. J. A. GUYER and M. C. TAYLOR, Assrs. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,617,305, 8.2.27. Appl., 19.4.26).—Gaseous mixtures containing chlorine are subjected to treatment with silica gel at an initial temperature not higher than 10°. H. ROYAL-DAWSON.

Purification of sulphur. T. GOLDSCHMIDT A.-G. (F.P. 608,930, 4.1.26. Conv., 17.1.25).—Crude sulphur is heated at a temperature sufficiently high to carbonise organic impurities, and is then filtered. L. A. COLES.

Manufacture of nitric acid and its salts. G. KASSNER (U.S.P. 1,616,900, 8.2.27. Appl., 24.3.24. Conv., 29.3.23).—See E.P. 213,571 ; B., 1925, 9.

Catalytic reactions [ammonia]. H. A. HUMPHREY, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,617,522, 15.2.27. Appl., 1.5.26. Conv., 4.5.25).—See E.P. 255,963 ; B., 1926, 821.

Manufacture of hydrogen. J. ROCHET, Assr. to COMP. DE PROD. CHIM. ET ELECTROMÉTALLURGIQUES ALAIS, FROGES, ET CAMARGUE (U.S.P. 1,617,965, 15.2.27. Appl., 17.2.25. Conv., 11.4.24).—See E.P. 232,184 ; B., 1925, 919.

VIII.—GLASS ; CERAMICS.

Glass-melting experiments with batch materials containing chemically combined water. E. M. FIRTH, F. W. HODKIN, M. PARKIN, and W. E. S. TURNER (J. Soc. Glass Tech., 1926, **10**, 364—373).—When, in batches for soda-alumina-silica glasses with 4—5% Al_2O_3, the alumina was supplied in the anhydrous form, melting was slightly quicker, working sweeter, and the glass less brittle than when hydrated alumina was used.

With soda-lime-alumina-silica glasses the hydrated alumina gave slightly better melting results, but the glass set more quickly and required a higher working temperature than glass from calcined alumina. When, for a soda-lime glass, crystallised sodium carbonate was used, melting was abnormal; a crust formed on the surface of the molten glass, which itself was very seedy, wavy, and cordy, and distinctly more viscous than the corresponding glass made from soda-ash. For glasses of the lamp-glass type, fused borax caused more rapid melting than either boric acid or crystallised borax, although the last-named caused the most rapid fining. None of the three forms possessed outstanding advantages as a source of boric oxide, but all were rather better than calcined borax. A. COUSEN.

Some glasshouse tests to determine differences in the general characteristics and in the working properties of molten glass. W. E. S. TURNER (J. Soc. Glass Tech., 1926, **10**, 374—384).—A series of detailed tests proved that an experienced glass-worker formed reliable views on the various working properties of the glasses handled, and deductions made by him were of distinct value in studying complex phenomena of melting and working. The tests confirmed the influence of moisture, arsenious oxide, and chlorides on the working properties of glass. A. COUSEN.

***X*-Ray patterns of mullite and sillimanite.** J. F. HYSLOP and H. P. ROOKSBY (J. Soc. Glass Tech., 1926, **10**, 412—415).—*X*-Ray photographs of natural sillimanite, mullite, kaolin (fired for 5 hrs. at 1050°), and a synthetic silicate obtained by heating alumina and silica in the proportions giving $3Al_2O_3,2SiO_2$ with 15% of sodium tungstate at 1350° for 100 hrs., revealed differences between the patterns of the sillimanite and the other samples examined. Since the differences depended on the relative spacing of the lines, they were sufficient for identification purposes. A. COUSEN.

Commercial sillimanite in glass works. A. COUSEN and W. E. S. TURNER (J. Soc. Glass Tech., 1926, **10**, 416—423).—The successful commercial use of sillimanite-clay refractories is cited in the case of a pot for opal glass, potettes, pot rings, port-hearth blocks, pot-furnace sieges, and coatings for flues. A. COUSEN.

Viscous properties of glass. V. H. STOTT (J. Soc. Glass Tech., 1926, **10**, 424—433).—The viscosity-temperature curve of a certain soda-lime-silica glass was perfectly definite and reproducible in the range 1200—1350°, if the glass had been rendered homogeneous by stirring at 1470°. On cooling below 1200° and reheating, the viscosity was not necessarily the same function of the temperature as before, and the glass could thus exist in more than one state at temperatures below 1200°. In the metastable range values of viscosity fluctuating by $\pm$ 30% from the mean were obtained. When the glass was not cooled below 1140°, the normal state was regained at temperatures slightly exceeding 1200°, but on slow cooling to the room temperature a persistent viscosity increase of 10% at temperatures above 1200° was obtained. Two different methods had shown that a change in sign of the curvature of the logarithmic viscosity-temperature occurred at about the same temperature as the discontinuity in the thermal expansion curve. The author's results did not confirm the formula given by Michelson (J. Geol., 1917, **25**, 405) for the return flow of glass after the removal of a load. A. COUSEN.

Causes of the surface devitrification of glasses. K. TABATA (J. Amer. Ceram. Soc., 1927, **10**, 6—22).—Tammann's explanation of the devitrification of glasses (cf. B., 1925, 920) is deemed inadequate, and the effects of sharp edges, cicatrices, and bubbles on crystal production at the surface layers of glasses are observed. Samples, the constitutions of which are reported and which had been carefully heated at a pre-determined temperature for a prescribed time, were subjected to microscopical examination. Surface devitrification not only depends upon constitution, but is remarkably stimulated by the presence of cicatrices and sharp edges. The explanation of the phenomena lies in the application of the conception of surface energy, there being a greater chance of chemical combination at the surface owing to the greater energy. Curvature of surface also influences the quantity of energy sustained in unit area, hence the effects of sharp edges and cicatrices. The action of surface energy is the most important factor in deciding the degree of devitrification on a glass surface. The paper contains many photomicrographs of the investigated effects. A. T. GREEN.

Heat transfer in [glass] furnaces. D. J. McSWINEY (J. Amer. Ceram. Soc., 1927, **10**, 1—5).—The factors influencing heat transfer in glass-furnace practice are discussed. Flame temperature depends not only on the character of the fuel, but also on its rate of combustion. The maximum temperature possible with a fuel is never attained in practice because intimate pre-mixing of the fuel with air never obtains in commercial furnaces. Flame temperature affects the rate but not the efficiency of heat transference. The completeness of heat transfer from flames does not appear to be affected by variations in luminosity. Convective heating and the circulation of gases are discussed. Rapid changes in the volume of the gases during and following combustion are important factors in promoting the circulation of gases. A. T. GREEN.

***X*-Ray analysis of clays.** L. B. STRUTINSKI (J. Russ. Phys. Chem. Soc., 1926, **58**, 314—325).—The *X*-ray examination of clays affords an accurate means of determining the relation between the size of the particles and the cohesive properties of the clay. The *X*-ray K_α (line of copper) photographs of a variety of Russian clays, both plastic and rigid, including pure kaolin, were investigated. The presence of comparatively large crystals of quartz was detected. The kaolin, which was coarse-grained even when ground, gave a characteristic diagram, some lines of which persisted in all the clays, which was due to its space-lattice. The constants of this were calculated, and several possible structures discussed. A symmetrical molecule, suggested by Samoilov, is adopted. The broadening of the lines in the *X*-ray photographs are measured, and the size of the particles calculated from these data. Most of the clay consists of particles larger than 10^{-5} mm., *i.e.*, the proportion of colloid is comparatively small, and the change in the *X*-ray diagram must be due to the deformation of the space-lattice. M. ZVEGINTZOV.

Substitution of calcined ball clay for non-plastic [materials] in porcelain and talc bodies. A. S. WATTS, R. M. KING, and H. G. FISK (J. Amer. Ceram. Soc., 1927, **10**, 53—58).—The effects of the substitution of properly-ground, calcined ball clay for (1) flint in a normal porcelain body, (2) talc in a normal talc body, (3) calcined kaolin in a normal chemical porcelain body, upon the modulus of rupture, resistance to impact and heat shock, and electrical resistivity, are reported. Recipes for the various bodies are given. The results obtained are variable and erratic. The only justification for the use of ordinary plastic ball clay in porcelain bodies is that this material imparts the necessary workable properties to the bodies; the quantity used should be reduced to a minimum.

A. T. GREEN.

Determination of the drying characteristics of clays. E. J. CASSELMAN (J. Amer. Ceram. Soc., 1927, **10**, 59—61).—Directions for laboratory tests for controlling the drying properties when new clays are substituted, and which can be used in conjunction with works' data, are reported. Three such tests are formulated, viz., (1) the measurement of the drying shrinkage; (2) the computation of the radius of curvature which results when the clay is dried in contact with a flat plaster mould; (3) the determination of the amount of cracking when a slip of definite consistency is dried in a flat plaster mould. As a guide, limiting values for these data are given.. A. T. GREEN.

Free quartz in felspar. A. MALINOVSZKY (J. Amer. Ceram. Soc., 1927, **10**, 23—29).—Felspars, which vary in composition, when used in the manufacture of ceramic wares, introduce variations in the coefficient of expansion of the products, due to the amount and nature of the quartz content. Petrographic examination not only shows the presence of this quartz, but also gives quantitative data agreeing with those obtained by the accurate calculation of a rational analysis from the proximate analysis. This shows that the calculated rational analysis can be of great service in controlling felspar additions. Photomicrographs of unfused and fused samples of felspar are given. A. T. GREEN.

Determination of mullite in porcelain. T. N. MCVAY (J. Amer. Ceram. Soc., 1927, **10**, 62—63).—A fired porcelain body in which mullite had developed, and a quantity of very pure, artificial crystals of mullite were subjected to treatment by (*a*) hydrofluoric, (*b*) sulphuric, (*c*) phosphoric acids in order to obtain quantitative data concerning the constitution of porcelain. The results obtained show that the use of hydrofluoric acid gives no indication of the amount of mullite present in porcelain, since the residues always contain quartz. Sulphuric and phosphoric acids are also unsuitable.

A. T. GREEN.

Effect of calcined cyanite in porcelain bodies. S. J. MCDOWELL and E. J. VACHUSKA (J. Amer. Ceram. Soc., 1927, **10**, 64—72).—The porcelain bodies compounded from clay, flint, felspar, and mullite, the last-mentioned being added as dissociated cyanite, were examined. The triaxial diagram is used to describe the composition of the bodies. The cyanite used consisted of 95% of cyanite, 2% of free quartz, and the remainder rutile and accessory minerals. This material was calcined at cone 18 for 2 hrs. to obtain complete dissociation. Screen analyses showed that over 85% of the flint, felspar, and calcined cyanite passed through a 260-mesh. The total clay content of the body, which was made up of 20% of Kentucky ball clay, 20% of Tennessee ball clay, 20% of Florida plastic kaolin, and 40% English china clay, was kept constant at 50% throughout the investigation. The clay slip was screened through 150-mesh, and the body de-watered on muslin in plaster absorption bowls to the proper working consistency. Firings of the experimental bodies were made in a gas-fired test kiln to cones 8, 10, 12, 14, 16, 18, and 20. The temperature was brought up to within three cones of that desired, and the bodies were soaked until the required cones bent over. Bodies containing the higher percentage of cyanite have the lower shrinkages and coefficients of expansion. The modulus of rupture tends to increase as the amount of cyanite increases at the expense of the felspar and flint, although 30% or more of cyanite must be present before the effect is marked. The colour of the bodies varied from good whites for the felspar-flint mixtures to greys for the cyanite mixtures. Microscopical examination showed that the dissociated cyanite particles are embedded in a ground-mass which has developed mullite from the clay, but that there is little if any secondary development of mullite. Those bodies high in cyanite had a protracted " maturing " range. A. T. GREEN.

Thermal conductivity of refractories. F. H. NORTON (J. Amer. Ceram. Soc., 1927, **10**, 30—52).—The main sources of error in previous investigations of the thermal conductivity of refractories are discussed. With a view of obtaining a perfectly linear flow of heat and an accurate measurement of temperature, the author details the design of an apparatus for determining the values of the " constant " up to 1600°, using a gas-fired furnace as the source of heat. The values of the thermal conductivity of a number of refractory materials, including fireclay, silica, magnesite, chrome, and carborundum products, are reported. It is believed that these values may have an error of $\pm$ 25%. The size and shape of the pores are important factors in influencing the value of the thermal conductivity. In most cases the conductivity increases rapidly at low temperatures and more slowly at high temperatures. Magnesite and silicon carbide, however, show the anomalous property of decreasing conductivity with increasing temperature. A rapid increase in conductivity at high temperatures is not indicated by the experimental results, probably because heat transfer by radiation does not become important at temperatures up to 1500°. A. T. GREEN.

Some corrosion and erosion phenomena and their bearing on the macrostructure of refractories. J. F. HYSLOP, R. GUMM, and H. BIGGS (J. Soc. Glass Tech., 1926, **10**, 405—410).—The corrosive and erosive attack of glass on refractory materials was simulated by exposing blocks of sealing-wax containing grog to the action of benzene. Corrosion depended on (1) the density of the solute-rich layer relative to the solvent and (2) the size of the grog particles. With a light, upward-flowing layer, material with large particles

was more rapidly broken up than that containing small particles, whilst the reverse held with a dense layer. The results were confirmed by immersions of clay rods in glass, clay with larger grog being more corroded than that with finer material in a lead glass, the reverse holding in soda–lime glass. When attack was due to erosion, maximum resistance coincided with the presence of the maximum amount of fine particles. A. Cousen.

Determination of the attack of slag and flue dust on refractories. F. Hartmann (Stahl u. Eisen, 1927, **47**, 182—186).—A cylinder of the refractory is heated in an electric carbon-granule furnace, and a definite quantity of solid slag is sprinkled on to it over a period of 30 min. The slag has time to react chemically and to run over the surface of the cylinder. The specimen is kept at the required temperature for a further period in order to give the slagged portion of the brick time to flow away. From the initial and final weight and volume of the refractory, the attack of the slag can be expressed as a percentage of the original weight or volume. A reducing or oxidising action can be employed. As the temperature rises above the m.p. of the slag, the rate of attack remains constant within a certain range, above which it increases. The effects of porosity, packing pressure, and surface sintering are considered. Comparison of the slagging action of iron oxide and manganese oxide shows that in the Siemens–Martin furnace slag the manganese oxide is the more active material. L. M. Clark.

Patents.

Colouring glassware. M. L. Burgess, Assr. to Marietta Manuf. Co. (U.S.P. 1,615,973, 1.2.27. Appl., 17.11.23).—To the batch in the furnace is added a suitable colouring medium to give a glass coloured throughout. The molten glass is rolled to the desired thickness and the hot surface sprayed with an aqueous copper sulphate–sulphuric acid solution, after which the glass is annealed, thereby baking-in the surface colour. A. Cousen.

Siliceous composition of matter. H. A. Endres and L. Caldwell, Assrs. to Celite Co. (U.S.P. 1,613,448, 4.1.27. Appl., 19.12.25).—Finely-divided silica, *e.g.*, kieselguhr, is mixed to a paste with an excess of lime and water, and when reaction is complete, is dried and calcined at 450—950°. The mixture of calcium oxide and calcium silicate thus obtained, on treatment with moist chlorine, yields a product which contains calcium hypochlorite dispersed through calcium silicate, and is of value as an ingredient of cement, or as a decolorising material for fats and oils. In an alternative process the calcination at high temperature is omitted. T. S. Wheeler.

Device for coating articles with glass, enamel, quartz, and metals. M. U. Schoop (U.S.P. 1,617,166, 13.1.22. Conv., 8.2.21).—The coating material in powder form is conveyed by a stream of combustible gas through a baffle plate, to attain a uniform suspension, to a nozzle supplied with oxygen and two combustible gases, forming a blowpipe flame. The coating material is projected from the nozzle on to the article to be coated, the particles being heated at a high temperature by the combustion of the conveying agent. B. W. Clarke.

Treatment of glass used in motor-car lamps etc. R. Hertog (F.P. 608,026, 16.12.25).—Glass is sprayed with a mixture of powdered rock having a high silica content, *e.g.*, quartz, with a binding agent, such as fluorspar, and heated to about 600° in a muffle furnace. L. A. Coles.

Annealing of glassware. Hartford-Empire Co., Assees. of V. Mulholland (E.P. 250,201, 13.3.26. Conv., 31.3.25).

Lehrs for annealing glassware. W. J. Mellersh-Jackson. From Hartford-Empire Co. (E.P. 264,446, 22.7.25).

Methods of and apparatus for forming sheet glass. L. Mellersh-Jackson. From Hartford-Empire Co. (E.P. 264,720, 31.5.26).

Manufacture of aluminium chloride (U.S.P. 1,617,695).—See VII.

IX.—BUILDING MATERIALS.

Apparatus for the thermal analysis of plaster. P. Jolibois and L. Chassevent (Compt. rend., 1927, **184**, 202—204).—As an adjunct to the chemical analysis of plasters an apparatus for thermal analysis is described in which a thermometer dips into a truncated, conical, brass vessel containing the paste, surrounded by a calorimeter-jacket containing water, the temperature of which is adjusted electrically. Curves relating the temperature-rise during setting and the time are given for a number of types of plaster. The apparatus enables control and classification of industrial plasters, according to their rates of hydration, to be obtained, by measuring the beginning of setting, the rate of transformation into gypsum, and the composition and resistance. J. Grant.

Patents.

Manufacture of cement. P. Cadre (E.P. 264,711, 7.5.26).—Gypsum, anhydrite, karstenite, or other materials containing a high proportion of calcium sulphate are dried to a moisture content of less than 5%, finely-pulverised, cooled, and mixed with dried and finely-pulverised, blast-furnace slags, clinkers, and the like, preferably in the proportion 80% of slag, 5% of clinkers, and 15% of anhydrite, or the like. The product, in contrast to ordinary cements of this type, is rapid hardening and possesses a high initial strength. B. W. Clarke.

Manufacture of fused cement. G. Polysius (G.P. 434,187, 19.9.24).—The materials, fused in the rotary kiln, are immediately run into a chamber in which an intimate mixing takes place, with the addition of a flux if necessary, the molten material being then subjected to a reduction process. The nozzle serving to conduct the heating medium is arranged so that the hottest part of the flame can reach the outlet of the kiln. The process can also be carried out on the counter-current principle. B. W. Clarke.

Manufacture of artificial stone. O. A. Tanner, Assr. to Artstone Burial Vault Co., Inc. (U.S.P. 1,617,927, 15.2.27. Appl., 25.7.25).—Stone is made from a mixture of 30 pts. of fireclay, 30 pts. of silica, 30 pts. of sulphur, 5 pts. of plumbago, and 5 pts. of talc. F. G. Crosse.

Wood-preserving composition. F. RASCHIG (U.S.P. 1,616,468, 8.2.27. Appl., 20.5.25. Conv., 13.2.25).—See E.P. 246,010; B., 1926, 241.

Siliceous composition (U.S.P. 1,613,448).—See VIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Relation of the mechanical properties of steel wire to the properties of the original wire and to the method of drawing. W. PÜNGEL (Stahl u. Eisen, 1927, **47**, 172—182).—The effects of cold-drawing on the mechanical properties of four steels, the carbon contents of which were 0·24, 0·41, 0·50, 0·62%, are considered. Ductility increases with decreasing carbon content, and the softer the steel the greater the amount of work it can withstand with safety. The tensile strength of both drawn and annealed wire increases proportionally to the carbon content. The ability to withstand bending and alternating torsion increases with increasing carbon content in wires of the same diameter and the same final strength. The tensile strength rises initially in proportion to the reduction in cross-section, but more rapidly with continued reduction. The change in bend strength follows that in tensile strength if the wire is bent over a constant radius. Bending tests carried out with a bending radius double that of the wire show a constant value for the number of bends sustained before fracture. Simple torsion tests, using a length of wire $l = 100 \times$ diameter, give results showing a constant or slightly falling strength in torsion with increasing reduction in area. Wires are examined for mechanical uniformity by twisting together two lengths laid side by side. This test gives constant values up to a certain reduction in cross-section, which is greater with smaller carbon content, and enables the limit to be fixed to which the material can be cold drawn. Endurance in the repeated torsion test falls with continued drawing; wires drawn from an original small diameter are superior to those drawn from a larger wire to give the same tensile strength. It is concluded that the higher carbon steels should be used for drawing wires which will be required to withstand repeated alternating torsion loading, since with higher carbon material a given tensile strength is produced with less cold work. The tests previously used for rope are not sufficient. The uniformity of the wire and its resistance to repeated alternating torsion should be examined.

L. M. CLARK.

Effect of electrolytic hydrogen on the tensile strength of steel in the presence of other elements. D. W. ALEXEEV and M. N. POLUKAROV (J. Russ. Phys. Chem. Soc., 1926, **58**, 511—517).—The time required for a given steel wire to break under a load of 3 kg. when acting as a cathode in solutions of potassium hydroxide and sulphuric acid under different conditions was investigated. In pure acid or alkaline solutions electrolytic hydrogen had no appreciable effect on the tensile strength of the steel, but small quantities of mercury in the alkaline solutions, or arsenic, antimony, and lead in the acid solutions, lowered it to about 1/30 of its original value. The magnitude of the lowering effect depended on the concentration of the metals, of the electrolytes, the current density, and the temperature. The effective concentrations of mercury were 0·03—0·125%, whilst arsenic was active even at 0·005%. The concentration of alkali below 0·25*N* and above *N* favour the lowering, as also increase of current density up to a maximum at 0·2 amp. per sq. cm. With polished wires, increase of temperature from 10° to 17° favours the lowering, but further rise inhibits it. Unpolished wires are unaffected at all temperatures above 10°. The action of arsenic, antimony, and lead is very similar to that of mercury. In all cases a surface deposit of the metal on the steel is formed, which promotes the action of the electrolytic hydrogen. A theory of the mechanism is suggested.

M. ZVEGINTZOV.

Influence of compression on the fragility of steel. Existence of a limit of fragility. P. DEJEAN (Compt. rend., 1927, **184**, 188—189).—The resilience of a steel containing 0·180% C, 0·265% Si, 0·54% Mn, 0·016% S, and 0·016% P has been determined, after annealing for various times at various temperatures. The resilience was then re-determined after the sample had been subjected to various forces of compression. In each case the pre-compression had no effect on the resilience until a critical value (ranging from 55—130 kg./mm.^{2}) was attained, when the latter fell rapidly.

J. GRANT.

Fluorspar flux in the iron foundry cupola furnace. E. WILKE-DÖRFURT and T. KLINGENSTEIN (Stahl u. Eisen, 1927, **47**, 128—133).—The use of fluorspar as a flux in the cupola furnace produces a slag which is less basic and less fluid than that obtained by using a pure limestone. This is ascribed to combination of the lime with silica and alumina. The slag from a charge mixed with spar contains no fluorine compounds, these probably being completely volatilised during the formation of the slag before the molten iron is covered by it. It is concluded that fluorspar can have no desulphurising action in the furnace, and that its use is harmful.

L. M. CLARK.

Testing moulds and mould materials. L. TREUHEIT and L. TREUHEIT (Stahl u. Eisen, 1927, **47**, 121—128).—The ramming strength, binding properties, and permeability to gas of sand moulds were determined for varying moisture and clay content. Various methods of mechanical and hand ramming were used. Ramming strength was measured by the size of indentation produced by a cone when pressed into the material under known load. Binding strength was determined by the ratio between the cross-section of a rectangular slab and the weight of that portion of the slab that could project unsupported without failure. All mechanical processes gave higher hardness values than hand ramming. High hardness was generally accompanied by flaws owing to unequal distribution of stresses throughout the material. The most useful hardness values were obtained with 7—10% of moisture and 8—12% of clay. In general, binding strength increased with resistance to indentation. L. M. CLARK.

Recovery of flue dust containing zinc and lead oxides from Siemens–Martin furnace flue gas. S. SCHLEICHER (Stahl u. Eisen, 1927, **47**, 169—172).—

Addition of scrap containing galvanised sheet, lead-bearing material, and enamelled iron to the charge of the open-hearth furnace produces considerable amounts of zinc and lead oxides in the flue gases. Adaptation of the Cottrell–Möller electrostatic precipitation method enables this dust to be recovered. Precautions are necessary against explosion due to admission of unburnt gas to the flue gas in the precipitation chamber during reversal of the furnace, or to pocketing of coal dust in sheet scrap in the bath with subsequent sudden combustion. The efficiency of the recovery process is increased by elimination of air leaks in the flue-gas leads and valves, and by lowering the temperature of the gases by water-spraying before their entry into the precipitation chamber. Lead and zinc resemble manganese in having a deoxidising and desulphurising action on the melt, whilst enamel is taken up by the slag at a very small expense of lime. The use of scrap containing tinplate may be more objectionable, since tin enters into the steel to an appreciable extent.
L. M. Clark.

Electrolytic working of brass cuttings in hydrochloric acid solution. W. W. Stender (Z. anorg. Chem., 1926, **159**, 145—157).—The difficulties of recovering copper electrolytically from brass on a commercial scale are discussed. The recovered copper must be such that it is readily dissolved by dilute sulphuric acid, and the zinc produced in a form other than zinc sulphate which is of little technical use. The possible methods of procedure are discussed and a solution of the problem is found in the electrolysis of brass in hydrochloric acid solution. A detailed account of the apparatus and process is given, and it is pointed out that (1) the yield for the current used was 88% of the theoretical, and (2) the energy expended was about 0·50 kw.-hr./kg. of copper precipitated in the form of powder. Zinc chloride was obtained as a useful by-product. The best results were obtained using ferro-concrete tubs with asphalt linings or wooden tubs with lead casings. The final product must be washed free from hydrochloric acid to prevent loss by oxidation. The course of the reaction is discussed theoretically, and an explanation put forward to account for the breaks in the curve reaction voltage–time, which occur towards the end of the reaction. M. Carlton.

Efficiency of ferrosilicon furnace. Schlumberger. —See VII.

Effect of slag on refractories. Hartmann.—See VIII.

Patents.

Producing corrosion-resisting coating on iron and steel. C. H. Humphries, Assr. to Metals Protection Corp. (U.S.P. 1,614,303, 11.1.27. Appl., 22.11.26; cf. 1,545,196; B., 1925, 726).—Iron and steel articles are protected against corrosion by coating them with a metal which is electropositive to iron (zinc), then with a metal which is electronegative to the first coating (copper) and finally with chromium. T. S. Wheeler.

Adherent, rust-proof, electrolytic metallic coatings on iron and steel. Langbein-Pfanhauser-Werke A.-G. (G.P. 433,037, 28.6.25).—Before the deposition of the final coating, two intermediate layers, the second of cadmium and the first of a metal easily alloying with cadmium (zinc, tin, or the like), are deposited on the iron. The coatings are readily polished and rust-proof.
L. M. Clark.

Tempering steel with hydrogen or gases containing hydrogen. Gelsenkirchener Bergwerks A.-G., Abt. Schalke (G.P. 431,622, 25.1.24).—A fixed upper limit of concentration (determined by the pressure and temperature) of methane produced in the reaction must not be exceeded, otherwise the reaction is retarded or stopped. L. M. Clark.

Method of increasing the ohmic resistance of pure iron, especially electrolytic iron. Siemens & Halske A.-G. (E.P. 245,753, 21.12.25. Conv., 9.1.25).—A very adherent deposit of silicon or boron is formed on the surface of pure iron by heating it at 600—700° in a current of a volatile compound of silicon or boron, *e.g.*, silicon hydride. This deposit is then made to diffuse into the iron by raising the temperature to 1000°, either in a current of hydrogen or *in vacuo*. To obtain a great increase in the ohmic resistance of the iron, the whole process may be repeated several times. The silicated layer on the iron may also be produced by embedding the iron in finely-pulverised silicon or in ferrosilicon rich in silicon, and heating at 1000°.
M. E. Nottage.

Alloy steel. C. M. Johnson, Assr. to Crucible Steel Co. of America (U.S.P. 1,617,334, 15.2.27. Appl., 27.6.25).—A steel containing 25—30% Cr, 20—30% Ni, and 1—3·5% Si. F. G. Crosse.

Copper-extraction process. W. E. Greenawalt (U.S.P. 1,614,668—70, 18.1.27. Appl., [a], 5.10.25. Renewed 20.7.26. [b], 10.4.26. [c], 8.6.26).—(a) The ore is finely ground in a relatively small amount of water, a dilute acid solution added to it, the resulting acid ore pulp graded to remove the oversize, which, after reducing its acid content, is added to new ore to be reground. (b) The copper ore is separated into a high-grade and a low-grade concentrate, the latter is roasted and leached with a dilute acid solution to extract the copper. The resulting solution, containing iron salts, is electrolysed, by which means the copper is deposited, and the acid and ferric iron are regenerated. The high-grade concentrate is fused and further concentrated, and added to the electrolysed solution to reduce the ferric iron. (c) The unroasted high-grade concentrate as above is added to the electrolysed solution to reduce the ferric iron. The solution is again electrolysed to deposit more copper. M. E. Nottage.

Production of stains and gold tones on brass. W. Stahlschmidt (G.P. 432,827, 10.7.24).—Brass is stained by treatment at ordinary temperatures with a bath of potassium hydroxide (168 pts.), sodium potassium tartrate (280 pts.), and copper sulphate (104 pts.), dissolved in water (3000 pts.). Immersion in dilute sulphuric acid produces a gold tone. L. M. Clark.

Granular brazing solder. C. H. Davis, Assr. to American Brass Co. (U.S.P. 1,617,285, 8.2.27. Appl., 21.7.26).—The solder consists of granules of a copper-zinc alloy coated with copper. M. E. Nottage.

Condensation of zinc vapours on heated metallic surfaces. Gelsenkirchener Bergwerks A.-G., Abt. Schalke and F. Caspari (G.P. 432,795, 11.2.22).—Zinc

vapours produced by the action of an electric current are deposited on metallic surfaces maintained at temperatures varying from 400—700°. Careful temperature control is necessary. L. M. Clark.

Zinc blende roasting furnace. G. Balz (G.P. 432,232 and 432,303, [A], 6.12.25. Addn. to 419,308, [B], 2.4.25).—(A) Introduction of secondary air takes place from above to the transmission opening in the inner cock. By means of a directing device, the air stream is thrown upon all the zinc blende as it enters. The tube for introducing the air is led in through the waste-gas exit, and is moveable. (B) The rotating stages are adjusted to move in opposite directions to produce homogeneous distribution of the material. Separation of the stages is effected by a sand trap formed from the roasted blende. L. M. Clark.

Separation of antimony and lead from mixtures of the oxygen compounds. A.-G. f. Bergbau, Blei- u. Zinkfabrikation zu Stolberg u. in Westfalen, and G. Darius (G.P. 433,031, 4.3.24).—The compounds are heated, in the absence of air and without fusion, with alkaline-earth sulphides or with materials from which these are produced during the process (such as a mixture of iron pyrites and alkaline earths). Sulpho-antimony compounds and insoluble lead sulphide are thus formed. L. M. Clark.

Treatment of impure molten metallic masses. H. Harris (E.P. 244,424, 17.10.25. Conv., 11.12.24).—Impure molten lead, containing one or more alloying metals, such as arsenic, antimony, or tin, is mixed with sodium hydroxide, with or without sodium chloride, heated at 500°, and an alkali oxy-salt of a lead-alloying metal gradually added. The lead-alloying metal must have less affinity for oxygen than the metal or metals to be removed from the lead. A reaction of the following type takes place:—$4NaSbO_3 + 6NaOH + 5Sn = 5Na_2SnO_3 + 3H_2O + 4Sb$. The metal set free in this reaction alloys with the lead. The alkali oxy-salt is used in conjunction with, or instead of, a commercial oxidiser, such as sodium nitrate, and it may be derived from the spent reagent used in a previous treatment. By this means the whole of the alkali can be utilised without separate recovery from the spent reagent, and the amount of commercial oxidiser needed is much reduced (cf. E.P. 142,315, 142,398, and 184,639; B., 1920, 456 A, 1922, 821 A). M. E. Nottage.

Heat treatment and concentration of ores. E. Edser, B. Taplin, and Metals Production, Ltd. (E.P. 264,584, 21.10.25. Cf. 250,991; B., 1926, 590).—The ore is heated in the presence of carbonaceous material and a halogen at a temperature such that the metal is reduced from the ore, and is subsequently separated from the gangue either by a physical concentration process or by leaching. The halogen employed is preferably chlorine, and is present in the form of a chloride of an alkali or alkaline-earth metal unless the ore itself contains compounds of these metals, in which case the chlorine should be added in the form of chlorides of manganese, iron, or copper. When calcite is present in the ore in large quantities it is advantageous to convert it into calcium sulphide by adding a metallic sulphide, *e.g.*, pyrites or chalcopyrite during the heat treatment. This process is applicable to ores containing lead or silver. M. E. Nottage.

Decomposition of cobalt ores. E. Schulze (G.P. 432,305, 21.8.24).—Ores containing cobalt as oxide or carbonate are decomposed by treatment with sulphurous acid under suitable conditions of temperature and pressure. Gangue is only slightly attacked by this reagent, so that the solutions obtained have a high degree of purity. L. M. Clark.

Separation of metals which form volatile compounds. Siemens & Halske A.-G. (G.P. 433,218, 17.2.24).—The mixture of volatile compounds is brought into contact with a gas stream the speed of which is adjusted so that the less volatile compound is carried away in the stream while the more volatile compound diffuses against the gas stream. The process is especially suitable for the separation of hafnium and zirconium. L. M. Clark.

Gold-saving device. H. A. Crosby (U.S.P. 1,616,784, 8.2.27. Appl., 25.1.26).—The machine consists of a separator inside which are two concentric cylinders mounted coaxially on a shaft. The outer cylinder is provided with a discharge spout, and connected with it, at the bottom, is a bowl containing mercury. The material to be separated is received in the inner cylinder. The shaft carries a partially spherical, agitating member placed within the bowl and formed with an opening for the passage of material through it. M. E. Nottage.

Silver alloy. W. L. Mitchell, Assr. to Tiffany & Co. (U.S.P. 1,614,752, 18.1.27. Appl., 14.8.24).—An alloy of silver with up to 10% Na is mixed with an alloy of silver and zinc to give a product containing 93% Ag, 6·5% Zn, and 0·5% Na, which does not form an adherent deposit on exposure to air. A small quantity of a hardening agent, *e.g.*, tin or antimony, can be added. T. S. Wheeler.

Apparatus for heat-treating metals and alloys. W. S. Smith, H. J. Garnett, and J. A. Holden (E.P. 263,923, 3.10.25).—The apparatus, designed for the annealing of electric cables etc., consists of a furnace, preferably electrically heated, inside which is a heating-tube through which the conductor is passed. This tube may be extended outside to form a cooling-tube which is water-jacketed. A portion of the tube within the furnace is perforated and jacketed, the jacket being provided with a pipe connected with a supply of inert gas; a second gas-supply pipe is fixed to the tube at a substantial distance apart from the jacket. Alternatively, the jacket may be replaced by a pipe which traverses a considerable length of the furnace before making connexion with the pipes attached to the heating tube. The gas entering the tube divides into two streams, one of which passes out into the atmosphere through the cooling-tube, the other flows through the heating-tube and escapes through the inlet end. The pressure of the gas supplied may be so adjusted that practically all the gas inside the jacket and that passing through the perforations into the heating-tube is discharged into the atmosphere at the inlet end, whereas the gas entering through the second supply-pipe passes out mainly through the outlet end. Thus the inert gas

is raised to the temperature of the conductor before coming into contact with it, and any traces of oil or grease become volatilised and are blown out into the atmosphere at the inlet end, whereby no reduction of copper oxide in the copper is possible.
M. E. Nottage.

Furnace for annealing sheet-metal plates. Stahlwerke A.-G., and W. Bernatzky (E.P. 264,110, 22.10.26).—The sets of materials to be treated are placed on portable supports by means of which they are inserted into and removed from the furnace. These supports are made longer than the inner furnace length so that their edges are not exposed to the direct furnace heat, and the coupling can be done during the annealing process outside the furnace. The furnace has two doors, and coupling and conveying contrivances are provided for the supports at each end of the furnace.
M. E. Nottage.

Annealing metal. H. W. Sanford (U.S.P. 1,613,726, 11.1.27. Appl., 11.2.22).—Castings are annealed by being placed in an enclosure through which air or other gas, *e.g.*, waste furnace-gas, at a suitable temperature is passed, so that the metal is slowly heated, and then slowly cooled. T. S. Wheeler.

Manufacture of chill castings. E. Kötteritzsch (E.P. 263,123, 6.12.26. Conv., 15.12.25).—The mould is provided at one side with a slot which extends nearly to the bottom, and which can be gradually closed by a series of independent movable pieces so arranged that, once closed, they do not move again in relation to the metal. The filling of the mould is thus effected by compartments, in each case through the aperture of the next higher compartment, which aperture is closed when this compartment is to be filled. The crucible, equal in content to the mould, is so guided that its inclination is automatically adjusted to the quantity of metal contained in it, and may be so mechanically associated with the closing pieces that as the casting progresses the next compartment to be filled is automatically closed. M. E. Nottage.

Fusion in a rotatory furnace. G. Polysius Eisengiesserei u. Maschinenfabr. (G.P. 432,511, 20.9.24).—In addition to coal dust or usual fuels, the heat from an electric current is used. One portion of the furnace is arranged as the fusion chamber, and into it the fuel and auxiliary heating are introduced. The process is especially suitable for the reducing treatment of melts.
L. M. Clark.

Rotatory roasting furnace. E. H. Kauffmann (G.P. 432,947, 11.3.24).—The front of the furnace is built up as a dust chamber. The ore is introduced in the second part directly behind the dust chamber, and ore dust beyond that. An economy in fuel is obtained.
L. M. Clark.

Determining the melting point of metals. G. Mantelet (G.P. 433,244, 21.12.24. Conv., 20.12.23).—The metal is melted in a crucible attached to one end of a fire-proof rod. The specimen is electrically connected to a warning device, and on the breaking of the circuit at the instant of melting a bell rings. A galvanometer, calibrated for temperature measurement, then indicates the fusion temperature. L. M. Clark.

Concentration of ores. J. L. Stevens (U.S.P. 1,614,089, 11.1.27. Appl., 1.7.26).—The addition of 0·1—1 lb. of an organic thiocyanate, *e.g.*, *n*-butyl thiocyanate, to 1 ton of a sulphide ore pulp greatly facilitates its froth flotation, especially in presence of an acid electrolyte. It is not necessary to isolate the thiocyanate, the liquid obtained by the interaction of *n*-butyl alcohol, potassium thiocyanate, and sulphuric acid can be employed. T. S. Wheeler.

Decomposing oxidisable ores by roasting. Soc. Metalurgica Chilena "Cuprum" (G.P. 412,134, 29.7.23).—The oxidising material is added to the ore in solid, liquid, or gaseous form in such manner and quantity that reduction to the metal is avoided. The pure metals are more difficult to separate from the ores than are the oxides. L. M. Clark.

Process of preparing metals. F. Koref and H. Hoffmann, Assrs. to General Electric Co. (U.S.P. 1,617,161, 8.2.27. Appl., 13.3.23. Conv., 7.8.22).—Metal wire consisting of a single crystal may be made by depositing metal of the same kind from a gaseous atmosphere on a single crystal, the deposit being conducted at a limited rate so as to avoid the formation of small crystals. M. E. Nottage.

Recovering metals from liquids and compositions therefor. A. L. Feild, Assr. to Electro Metallurgical Co. (U.S.P. 1,617,206, 8.2.27. Appl., 20.6.22).—The liquid is brought into contact with activated carbon having an iodine adsorption value in excess of 20%.
H. Royal-Dawson.

Recovery of zinc and zinc compounds. E. C. R. Marks. From American Smelting & Refining Co. (E.P. 265,036, 7.4.26).—See U.S.P. 1,605,641; B., 1927, 114.

Means for sintering fine ores or pulverous ores. H. G. Torulf (U.S.P. 1,618,116, 15.2.27. Appl., 30.10.24. Conv., 22.8.23).—See E.P. 220,924—5; B., 1924, 985.

[Dismantling rabble-arms of] pyrites and other furnaces. E. Bracq (E.P. 264,411, 27.8.26. Conv., 7.4.26).

Coating articles with metals (U.S.P. 1,617,166).—See VIII.

XI.—ELECTROTECHNICS.

Effect of moisture on electrical properties of insulating waxes, resins, and bitumens. J. A. Lee and H. H. Lowry (Ind. Eng. Chem., 1927, 19, 302—306).—Measurements of the dielectric constant, effective conductivity at 1000 cycles, and resistivity were made with thirty-one waxes, resins, and bitumens, natural and synthetic, in the initially thoroughly dry condition and after six months' immersion in a salt solution corresponding to exposure to 98% relative humidity. In general, increase in capacity and conductivity and decrease in resistivity were greatest with those materials absorbing the most water. The substances least affected and absorbing least water were of the hydrocarbon type (paraffin wax). All the materials examined absorbed moisture, the most marked absorptions being as follows: bayberry wax (6·1%), blown gilsonite and residual

petroleum oil (3·3%), aluminium stearate (2·7%), zinc stearate (3·0%), stearic acid (3·2—4·0%), and shellac (4·3—5·1%). C. O. Harvey.

Efficiency of carbide and ferrosilicon furnaces. Schlumberger.—See VII.

Patents.

Thermo-couples and thermo-elements. W. C. Heraeus G.m.b.H., and O. Feuszner (E.P. 264,201, 7.9.25).—One wire of a thermo-electric couple consists of an alloy of at least three precious metals, including a gas-adsorptive precious metal, *e.g.*, palladium, together with a precious metal of Group I of the periodic system, *e.g.*, gold, and one other precious metal of the platinum group, *e.g.*, platinum, in relatively small proportion. The other wire of the couple may be composed of platinum or platinum-rhodium etc. Couples so constructed yield constant values of thermo-electric potential when exposed to definite temperatures.

J. S. G. Thomas.

Electric discharge tube. N. V. Philips' Gloeilampenfabr. (E.P. 250,928, 6.4.26. Conv., 18.4.25).—In an electric discharge tube adapted more especially for guarding against excessive voltages, one or both electrodes consist partly of a mixture of an alkali compound, *e.g.*, sodium chloride, placed in a cup-shaped portion of the electrode, and a substance, *e.g.*, magnesium, capable of liberating the alkali metal from the compound. The cup-shaped portion of the electrode is preferably closed by metal gauze. If desired, the electrodes, *e.g.*, of nickel, may be amalgamated. J. S. G. Thomas.

[Magnesium electrodes] for electric discharge tubes. N. V. Philips' Gloeilampenfabr. (E.P. 249,847, 15.3.26. Conv., 28.3.25).—Magnesium is volatilised in a discharge tube and caused to deposit upon a conducting layer, *e.g.*, composed of graphite emulsion, coating the leading-in wire and the inner surface of the tube in the neighbourhood of the wire. J. S. G. Thomas.

Introducing potassium, caesium, or rubidium into electric discharge tubes. H. Wade. From N. V. Philips' Gloeilampenfabr. (E.P. 264,258, 22.10.25 and 20.1.26).—A mixture of one or more compounds of potassium, caesium, or rubidium, *e.g.*, caesium chloride, with one or more azides of the metals of the second principal group of the periodic system, preferably barium azide, is introduced into the discharge tube or into a receptacle connected therewith, and heated, whereby the metallic vapour of potassium, caesium, or rubidium spreads within the tube without risk of oxidation.

J. S. G. Thomas.

Electric storage batteries. C. E. Linebarger (E.P. 264,205, 17.9.25).—A storage battery is provided with channel ways communicating with the main part of the battery and containing hydrometric units, *e.g.*, small pellets or balls, for indicating the sp. gr. of the electrolyte.

J. S. G. Thomas.

Electric gas-filled discharge device. G. Holst, E. Oosterhuis, and J. Bruijnes, Assrs. to N. V. Philips' Gloeilampenfabr. (U.S.P. 1,617,684, 15.2.27. Appl., 8.2.22. Conv., 9.2.21).—See E.P. 175,275; B., 1923, 762 a.

Electric induction furnaces. J. R. de Zubiria (E.P. 251,608, 15.4.26. Conv., 29.4.25).

Electrodes for electric furnaces. Norske Aktieselskab for Elektrokemisk Ind. (E.P. 252,153, 6.5.26. Conv., 15.5.25).

Electric discharge devices. British Thomson-Houston Co., Ltd., Assees. of H. D. Brown (E.P. 253,126, 3.6.26. Conv., 3.6.25).

[Separators for the plates of] electric storage batteries or electric accumulators. Fuller's United Electric Works, Ltd., and S. C. Whaite (E.P. 265,000, 18.1.26).

[Plate pillars for] electric storage batteries or electric accumulators. Edison Swan Electric Co., Ltd., and G. E. Webster (E.P. 264,970, 25.11.25).

Asbestos (E.P. 241,576).—See VII.

Heat-treatment of metals [cables] (E.P. 263,923).—See X.

Alcohol from sugar (U.S.P. 1,612,361).—See XVII.

XII.—FATS; OILS; WAXES.

Theory of the hardening of fats. H. P. Kaufmann and E. Hansen-Schmidt (Ber., 1927, **60**, [*B*], 50—57).—Periodical determination of the iodine and thiocyanogen values during the course of mild technical hydrogenation of arachis oil shows that oleic acid is not reduced, whereas the main part in the change is played by linoleic acid, which is converted into singly unsaturated acids solid at the atmospheric temperature; the proportion of saturated components remains unchanged. More prolonged hydrogenation of sunflower oil causes an increase in saturated compounds at the expense of linoleic acid, the bulk of which is, however, transformed into singly unsaturated acids. Subsequently, reduction of oleic acid and of its intermediately formed isomerides commences. Simultaneously, displacements of the double linking occur (recognised by the discrepancy between the iodine and thiocyanogen values) which are due to dehydrogenation in an unexplained manner.

H. Wren.

Solidification point of edible fats. T. Meyer (Z. Unters. Lebensm., 1926, **52**, 461—465).—The method of Mohr (Milchw. Forsch., 1923, **1**, 15) for determining solidification curves of fats was found reliable for use in the margarine industry. The curves obtained were characteristic for the different fats. A good margarine mixture showed a solidification curve closely resembling that for butter. A. G. Pollard.

Rapid method for the sorting of butters and margarines. C. H. Manley (Analyst, 1927, **52**, 67—72).—The method depends on the butyric acid content, and a new value "*M*" is obtained which ranged from 20·6—26·4 for pure butters (20 samples) and 0—5·1 for margarines (18 samples). 5 g. of filtered fat are saponified with 20 c.c. of glycerol-soda solution (900 c.c. of glycerol and 100 c.c. of 50% aqueous sodium hydroxide solution), the soap is dissolved in 100 c.c. of boiled distilled water, and 4 drops of 0·5% methyl orange solution are added to the cooled solution, and 25% by vol. of sulphuric acid run in till the solution is faintly pink. The total volume of solution and precipitated acids is taken, 100 c.c. are filtered off, nearly neutralised with 10% sodium hydroxide solution, and neutralisation is

completed with $0 \cdot 1N$-sodium hydroxide. Phenolphthalein solution is then added and the soluble fatty acids are titrated, and the number of c.c. taken less that required for the blank is the "*M*" value. If to the neutral solution from a butter fat 0·5 g. of silver sulphate is added and a Kirschner value determined, the result (*KM*) is the same or very nearly so as the "*M*" value, it being probable that volatile soluble acids other than butyric acid are co-precipitated with the insoluble acids. For margarines the "*KM*" value is lower than the "*M*" probably owing to some caprylic acid escaping co-precipitation. Precipitation is complete in 5 min. Butter fat cannot be determined by this method in the presence of 5% of coconut oil, but boric acid has no influence. D. G. Hewer.

"Hydrogenation" of oleic acid with activated hydrogen. II. I. Waterman and S. H. Bertram (Chem. Umschau, 1927, **34**, 32—35; cf. Eichwald, A., 1922, i, 982).—The so-called hydrogenation of oleic acid by means of activated hydrogen (Bonhoeffer, A., 1925, ii, 52) has been critically examined. In a series of experiments the iodine value of oleic acid so treated never fell below 78, the refractive index rose consistently, and no appreciable alteration in elementary composition could be detected. The view that no hydrogenation takes place, but that the reaction is essentially a polymerisation, in the sense of two mols. of oleic acid giving rise to one mol. of a saturated dibasic acid, is supported by m.p. and mol. wt. evidence. E. Holmes.

Examination of mixtures of coconut oil and palm-kernel oil. Determination of butter fat in margarine. G. D. Elsdon and P. Smith (Analyst, 1927, **52**, 63—66).—The Reichert-Meissl and Polenske values have been determined for a complete range of mixtures of coconut and palm-kernel fats. From a consideration of the Reichert–Meissl, Polenske, and Kirschner values of a given mixture the proportion of butter fat is deduced, and the Reichert–Meissl and Polenske values due to it are allowed for; from the resulting figures the proportion of coconut and palm-kernel fats is deduced from the table. Accuracy depends upon the factors of the oils (chiefly of the butter fat) used in the mixture. Different mixtures of coconut and palm-kernel fats in certain proportions may give identical results. A table showing percentages of butter fats in margarines of varying Polenske and Kirschner values is included.
D. G. Hewer.

Oxidation and hydrolysis of light wood oil. P. O. Powers, A. Lowy, and W. A. Hamor (Ind. Eng. Chem., 1927, **19**, 306—308).—Fractions of light oil boiling below 195° obtained during the industrial distillation of hardwood (maple, beech, birch, ash, and oak), gave good yields of organic acids upon oxidation, the best results being obtained by boiling with 6% nitric acid. Vanadium pentoxide catalysed the reaction and diminished side reactions. Hydrolysis (by nitric acid) of the esters present in the oil takes place at 70°; at 80° the oil is oxidised. Hydrolysis of the oil by refluxing with lime and water gave methyl alcohol and calcium salts of acetic, propionic, butyric, and higher acids. No traces of alcohols other than methyl alcohol were found, but this contained appreciable amounts of ketones, chiefly methyl ethyl ketone. Removal of aldehydes and ketones from the original oil by means of sodium bisulphite caused a diminution in volume of 40%.
C. O. Harvey.

Effect of moisture on waxes etc. Lee and Lowry. —See XI.

Milk fat in foodstuffs. Lührig.—See XIX.

Salts of linolenic hexabromide from lumbang oil. Imperial and West.—See XX.

Patents.

Bleaching fatty oils, mineral oils, etc. H. Bollmann (E.P. 245,745, 2.12.25. Conv., 6.1.25).—The bleaching of oils by bleaching earth is accomplished most quickly and effectively if oil and bleaching agent be intimately mixed in optimum proportions, saturation of the bleaching earth with oil already bleached being avoided. An apparatus is described wherein fresh portions of preheated oil and bleaching earth are continually brought into contact in requisite proportions, and removed from the bleaching chamber.
S. S. Woolf.

Purification of dirty soap- and soda-solutions. A. Stern (Austr. P. 103,708, 5.2.25).—Dirty wash-liquors containing soap and soda are treated with a sufficient weight of alkali carbonate to restore their original alkali content, and the dirt is removed by filtration, preferably through spodium. S. S. Woolf.

[Soap for] cleaning fabrics (U.S.P. 1,615,146).—See V.

Siliceous composition (U.S.P. 1,613,448).—See VIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Physical concepts on problems in the paint and allied industries. A. de Waele (J. Soc. Dyers and Col., 1927, **43**, 42—47).—The permeability of an oil-containing protective coat of paint is inversely proportional to its degree of saturation (oxidation), and results from the presence of a polar group (relative to the oil) capable of lowering the contact angle of the coating to water. The property of paint by which it flows (deflocculates) during brushing and stiffens (reflocculates) after brushing, is dependent on the fact that whilst static rigidity is a function of the solid–liquid interfacial tension into the absolute surface of the interface, the rate of regain of rigidity after shear (rate of reflocculation) is an inverse exponential function of the viscosity of the medium $dF/dt = nfe^{-n}$, where F = the rigidity at any stage of deflocculation, n = viscosity of the medium, f = true static rigidity (rigidity after infinite rest), and e = log. base; thus with a unit increase in the viscosity of the medium the rate of reflocculation diminishes at a tenfold rate. The "solidity" or rigidity of an oil-bearing coating is a function of heterogeneous grading of the pigment present. The drying of printing ink on paper is mainly determined by the rate of deflocculation of the ink and its absorption by the paper material; the rate of penetration of paper by an ink is proportional to the viscosity of the medium. It is necessary to introduce suitable pigments into cellulose nitrate

lacquers for the purpose of inhibiting destruction of the cellulose nitrate by incident ultra-violet light.
A. J. HALL.

New paint and varnish remover. M. PHILLIPS and M. J. GOSS (Chem. Met. Eng., 1926, **33**, 745).—*p*-Cymene mixed with ethyl or methyl alcohol etc. forms an excellent solvent for the removal of paint or varnish. *p*-Cymene has the advantage over other solvents that it has a high b.p. (176—176·6°). (See U.S.P. 1,580,914; B., 1926, 554.) S. PEXTON.

Degree of saturation of the resin acids. I. B. M. MARGOSCHES, K. FUCHS, and W. RUZICZKA (Chem. Umschau, 1927, **34**, 29—32).—Mainly introductory. After discussing the methods previously used for determining the iodine values of resins, it is demonstrated that the rapid method previously developed (Margosches, Hinner, and Friedmann; B., 1924, 639) is applicable to the examination of the resin acids. In this method 0·10—0·11 g. of substance is dissolved in 10 c.c. of 96% alcohol and mixed with 25 c.c. of 0·2*N*-alcoholic iodine solution and 200 c.c. of water, the time of reaction being 5 min. Tables are given showing (*a*) the effect on the observed iodine value of varying amounts of iodine in excess of that required for saturation, whereby the 5 min. method gives results comparable with those from the Hübl 24 hr. method only when this excess is approximately 75%, and (*b*) comparative iodine values by the Hübl and Margosches methods for a series of thirteen resinous materials. E. HOLMES.

Esters of Congo and Manila resins. W. T. PEARCE, R. CARLSON, and C. L. RYDSTROM (Ind. Eng. Chem., 1927, **19**, 285—286).—The high acid values developed by Congo and Manila resins upon heating until oil-soluble cause "livering" in pigmented varnishes. Varnishes of low acid value may be produced from these resins, esterified by heating with glycerol at temperatures up to 325°, with or without the addition of catalysts. The acid value may also be reduced by heating and driving off volatile matter. C. O. HARVEY.

Effect of moisture on resins etc. LEE and LOWRY. —See XI.

Pigment finishes [for leather]. VOGEL.—See XV.

PATENTS.

Composition of cellulose ethers and esters with hygroscopic substances. [Flexible films.] C. DREYFUS and G. W. MILES, Assrs. to AMERICAN CELLULOSE & CHEMICAL MANUF. CO., LTD. (U.S.P. 1,611,169, 21.12.26. Appl., 27.12.20).—The addition of zinc chloride or similar inert hygroscopic substance (2—10 pts.) to a solution of a cellulose ether or ester, *e.g.*, cellulose acetate (100 pts.) in acetone, enhances the flexibility of the film formed when the solvent is evaporated in thin layers, provided the film is kept slightly moist, until the hygroscopic substance has absorbed such water as it can retain. T. S. WHEELER.

Cellulose-ester composition. R. H. VAN SCHAACK, JUN. (U.S.P. 1,612,669, 28.12.26. Appl., 21.8.25).—Benzyl esters of hydroxy-aliphatic acids, *e.g.*, benzyl lactate, tartrate, or citrate, are of value as blending agents for cellulose esters and alcohol-soluble resins.
T. S. WHEELER.

Making a phenolic resin varnish. L. M. ROSSI, Assr. to BAKELITE CORP. (U.S.P. 1,613,724, 11.1.27. Appl., 15.12.24; cf. U.S.P. 1,146,299—330; B., 1915, 914).—Phenol (1 pt.), formaldehyde (0·9 pt.), and sodium hydroxide (0·03 pt.) are boiled together in aqueous solution for ½ hr., powdered rosin (0·38 pt.), and then hexamethylenetetramine (0·091 pt.), in concentrated solution are added, and the mixture is cooled, and employed as a varnish to coat papers, in the manufacture of moulding mixtures, and, generally, as a substitute for alcoholic solutions of phenol resins.
T. S. WHEELER.

Manufacturing plastic compositions [artificial mother-of-pearl]. Obtaining coloured, polymerised styrene and its homologues and products [artificial amber]. I. OSTROMISLENSKY, Assr. to NAUGATUCK CHEMICAL CO. (U.S.P. 1,613,673—4, 11.1.27. Appl., [A], 29.1.25; [B], 3.2.25; cf. E.P. 236,891; B., 1925, 771).—(A) 1—10% of balata, rubber, or like gum is dissolved in styrene, with or without addition of a common solvent, *e.g.*, xylene, and the styrene is polymerised by heating at 135—140°. The product has a nacreous lustre similar to that of mother-of-pearl, and is less brittle than untreated polymerised styrene. (B) 0·5—1·0% of an azo dye is dissolved in styrene, which is then polymerised by heat. The product is ground and added to styrene, which on polymerisation yields a substance resembling amber. T. S. WHEELER.

Hardening phenol–urea formaldehyde products. H. V. POTTER, J. W. CRUMP, and DAMARD LACQUER CO., LTD. (E.P. 264,601, 23.10.25).—The syrupy condensation-products described in E.P. 256,711 (B., 1926, 889) can be hardened and eventually converted into an insoluble and infusible form by the addition, in the cold, of acids or acid-producing substances, *e.g.*, salts of strong acids and weak bases. The rate of hardening depends on the phenol–urea ratio and the accelerator used in the preparation of the original syrup, as well as on the nature and amount of hardening agent used in the present hardening process. S. S. WOOLF.

Preparation of condensation products from carbamide, or its derivatives, and formaldehyde. Reconversion of insoluble carbamide–formaldehyde condensation products into soluble form. GES. FÜR CHEM. IND. IN BASEL (F.P. 609,108—9, 11.1.26. Conv., 17.1.25).—(A) According to the duration of heating and the temperature employed, water-soluble, water-insoluble, or gelatinous condensation products are obtained when carbamide or its derivatives and formaldehyde are condensed together under increased pressure and at temperatures above 100°. The products are used for insulating varnishes, moulding compositions, etc. (B) Splinters or borings from hardened carbamide–formaldehyde condensation products are heated under increased pressure and at above 110° with excess of formaldehyde. The latter is subsequently distilled off or further condensed with an appropriate amount of carbamide. A soluble resin-syrup is obtained in each case, and can be used for the same purposes as is the original resin before hardening. S. S. WOOLF.

Preparation of artificial resin. CONSORTIUM FÜR ELEKTROCHEM. IND. G.M.B.H., Assees. of H. DEUTSCH

W. Haehnel, and W. O. Herrmann (G.P. 433,853, 10.8.22. Addn. to 422,538).—A mixture of acetaldehyde and mesityl oxide is heated under reflux with dilute sodium hydroxide solution, distilled, and heated at 150°, and the resinous residue is ground with 3% acetic acid. The resin powder is separated, washed, and heated at 200°, yielding elastic, alcohol-soluble, light-coloured, and easily-fusible resin that can readily be rolled out to laminæ. Benzophenone may be used as solvent during the heating of the resin at 150°. S. S. Woolf.

Manufacture of paints. D. Gardner (U.S.P. 1,617,754, 15.2.27. Appl., 21.8.22. Conv., 11.10.21).—See E.P. 195,996; B., 1923, 564.

Preserving specimens (E.P. 263,674).—See I.

Ester of *n*-butyl alcohol (U.S.P. 1,613,366).—See XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Accelerators of vulcanisation: their influence towards one another and towards various rubber-compounding ingredients. R. Ditmar (Gummi-Ztg., 1927, **41**, 992—994, 1048—1050).—A comparison of the behaviour of a series of accelerators of vulcanisation alone and in the presence of various common ingredients for rubber, using a basic mixture of rubber (100 pts.), sulphur (6 pts.), and zinc oxide (20 pts.). It is found that the effects produced are not explicable by any simple chemical law, so that it is necessary to investigate independently for each accelerator the behaviour under different conditions. White and brown factice, lithopone, and whiting impair the activity of the accelerators, the degree, however, being different in each case and for different accelerators; the direction of the effect of barytes is dependent on the accelerator; kieselguhr, lime, magnesium oxide, and magnesium carbonate intensify the accelerating effect.
D. F. Twiss.

Classification and testing of regenerated rubber. P. Alexander (Kautschuk, 1927, 67—70).—The chief forms of old rubber serving as stock for reclaiming or regenerating, in decreasing order of magnitude, are motor covers, inner tubes, and rubber shoes. Distinction is necessary between "pure" and "mixed" reclaimed rubber; during the manufacture of the latter extraneous compounding ingredients have been incorporated. For chemical examination of reclaimed rubber the determination of acetone extract, saponifiable matter in the residue after extraction, bituminous ingredients, and ash suffice for practical purposes. A vulcanisation test, *e.g.*, for 20 min. in steam at a pressure of 3 atm. after the addition of 3% of sulphur, is particularly valuable, consideration being given both to the tensile strength and the ultimate elongation of the product.
D. F. Twiss.

Sedimentation analysis of fillers (for rubber). F. Evers (Kautschuk., 1927, 70—77).—A description of the Wiegner–Gessner sedimentation apparatus (A., 1926, 377), and its applicability to the investigation of the relative proportion of particles of various sizes in powders intended for incorporation in rubber. Results are quoted of experiments with golden antimony sulphide, magnesium carbonate, magnesium oxide, gas black, zinc oxide, sulphur, litharge, and lead sulphate.
D. F. Twiss.

Patents.

Method of treating rubber and the like, and the products obtained thereby. J. H. Reel, Assr. to General Rubber Co. (U.S.P. 1,612,780, 28.12.26. Appl., 20.6.24).—Dry rubber obtained in an uncompact plastic form by an atomising treatment is subjected to a process removing non-rubber constituents and yielding purified rubber in a similar uncompact condition.
D. F. Twiss.

Rubber-latex coagulum. A. H. Petersen, Assr. to Kelly-Springfield Tire Co. (U.S.P. 1,611,278, 21.12.26. Appl., 26.9.22).—The coagulant (1 pt. by wt.) dispersed in a liquid medium (5 pts.) is mixed with rubber latex (containing 2 pts. of rubber); the mixture is agitated and the resulting coagulum is removed from the liquid. D. F. Twiss.

Process of softening rubber. C. P. Hall (U.S.P. 1,611,436, 21.12.26. Appl., 23.7.25).—In order to facilitate the milling of rubber there is added a softening agent with the following characteristics: Minimum solubility in carbon disulphide, 99%; in carbon tetrachloride, 98%; in hexane, 75%; minimum $d^{15\cdot5}$ 1·06, d^{25} 1·05. Maximum fixed carbon 14%, paraffin scale 2%, flash point 190°, evaporation loss 3·0% on 50 g. in 5 hrs. at 162·5°. Engler viscosity 5—7.
D. F. Twiss.

Production of a water-soluble evaporation product from [rubber] latex. M. S. Stutchbury, Assee. of W. Bachmann and F. Hebler (G.P. 432,894, 30.9.23. Addn. to 419,658).—The effectiveness of the protective colloids in the evaporation of latex is raised by the addition of hydrocyclic compounds such as tetra-, hexa-, methylhexa-, and deca-hydronaphthalene, *cyclo*hexanol, etc. D. F. Twiss.

Method of treating balatas and gutta-perchas. C. H. Smith, Assr. to Goodyear Tire & Rubber Co. (U.S.P. 1,614,541, 18.1.27. Appl., 13.12.24).—Balata or gutta-percha is repeatedly treated with a solvent progressively lower in resin content for the removal of its natural resins; the de-resinised product is dissolved in fresh solvent and treated with an inert decolorising agent, which is then removed, the balata or gutta product being finally precipitated. D. F. Twiss.

Chemical process for the etching of rubber. E. Sardou (U.S.P. 1,614,935, 18.1.27. Appl., 29.9.25. Conv., 15.10.24).—The lines of the design reproduced on rubber sheets, which have been subjected to superficial super-vulcanisation, are covered with an impalpable, powdery mixture of fatty and resinous substances; the surface of the sheet is then subjected to the action of a solution of nitric acid and potassium bichromate.
D. F. Twiss.

Method and apparatus for uniting crude rubber to leather or other supporting surfaces. E. F. H. Enna (E.P. 247,929, 26.1.26. Conv., 21.2.25).—A layer of raw rubber is united with a supporting surface of leather, rubber, or other material by roughening the surface of the former, and immediately, without the application of chemicals, pressing it into close contact

with the supporting surface. Two supporting surfaces can be similarly joined by pressing together with a freshly-roughened, intermediate uniting layer of raw rubber. Suitable apparatus is described.

D. F. TWISS.

Use of mono- and di-carbalkoxydiarylthiocarbamides as accelerators in the vulcanisation of rubber. BRITISH DYESTUFFS CORP., C. J. T. CRONSHAW, and W. J. S. NAUNTON (E.P. 264,682, 1.3.26. Cf. 244,159; B., 1926, 138).—The reaction product obtainable by treating an alkyl anthranilate with thiophosgene, *e.g.*, di-*o*-carbethoxydiphenylthiocarbamide, or the further product of heating this at vulcanising temperatures, is used as an accelerator of vulcanisation.

D. F. TWISS.

Process for vulcanising rubber. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 264,673, 15.2.26).—The condensation products of crotonaldehyde or of an αβ-disubstituted acraldehyde with an aromatic amine are applied as accelerators of vulcanisation. D. F. TWISS.

Manufacture of new condensation products of substituted acroleins. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 264,674, 15.2.26).—Aromatic amines react with αβ-disubstituted acraldehydes with formation of condensation products. Suitable solvents or condensation agents may be used if desired (cf. preceding abstract). D. F. TWISS.

XV.—LEATHER; GLUE.

Influence of the degree of liming on the amount of tannin absorbed by the hide. K. H. GUSTAVSON and P. J. WIDEN (Collegium, 1926, 562—569).—Pieces of pelt were limed for different periods, unhaired, washed, delimed with ammonium chloride solution, washed, preserved for 12 hrs. in a boric acid solution, washed with distilled water, treated with a sodium-acetic acid buffer p_H 5·0, washed, cut into small pieces, and submitted to various tanning tests with basic chromium sulphate solutions of 22%, 37%, 54%, and 60% basicities, and an anodic oxalato-chromium solution. The amount of chromium absorbed by raw hide and the other pieces from the different chrome liquors showed that the longer the liming up to 2 weeks, the greater is the amount of chromium absorbed. The percentage increase was greater in the more basic liquors. This was expected, and was due to the action of the colloidal constituents and the anodic complexes, which action increases with the basicity. The basicity of the chromium salt on the fibres of raw hide was much less than that of the salt on the fibres of the limed pieces of pelt. The basicities of the salts fixed by raw hide from 22% and 37% liquors were 15% and 36·2%, respectively, lower than that of the original liquor used for tanning. Hide powder and limed pelt fixed a more basic salt from these chrome liquors. This might be attributable to a stronger activation of the acid groups due to salt formation of lime and soda with the collagen. If this were so, it would agree with the view that cathodic complexes are taken up to a much greater extent from liquors of greater basicity (65—75%) than the hydrolysed acid. It was found that just as much chromium was fixed if the basic groups were inactivated. The increase in the amount of tannin absorbed, due to liming the pelt, is due just as much to colloidal changes as to the activation of the protein groups. Samples of hide powder were treated with acids and alkalis, respectively, then brought to the isoelectric point, and their behaviour towards different tanning materials determined and the pelt tests confirmed. Hide which had been one-third peptised by treatment with trypsin showed diminished chromium absorption. The colloido-chemical conception of tanning can explain the formation of high molecular compounds just as well as the chemical theory, and both theories should be combined. The experiments show that even raw hide possesses a great affinity for those basic chromium compounds in which the colloidal nature is not too pronounced. It is still doubtful whether the ring structure of the raw hide is opened to liberate some auxiliary valencies to combine with the hydrolysed acid, or whether the ring structure itself has the power to combine directly with the chromium salts. Another possibility is that the elementary components possess free acid and basic groups. Liming results in the formation of a chemical compound with hide protein involving primary valencies. This leads to a splitting up into the component parts, thus increasing the acid and basic groups. Salt formation causes a new orientation of the auxiliary valencies owing to new structural arrangements, and there is consequently an increase in the auxiliary valencies. The aggregates of elementary constituents which had formed by cohesion, residual valencies, and similar forces are broken up by liming.

D. WOODROFFE.

Influence of temperature on the leaching of oak wood, willow bark, pine bark, and badan root. P. PAWLOWITSCH (Collegium, 1926, 570—571).—Oak wood, willow bark, pine bark, and badan root have been leached at different temperatures, respectively, and the leach liquor was analysed in each case. The leached tannin increased with increase of temperature to a maximum and diminished with further increase of temperature in the case of willow bark and badan root. The best temperature for the extraction of oak wood was 90—100° and higher up to 121°, for pine bark 90—100°, for willow bark 64—70°, and badan root 70—80°. The optimum temperatures for leaching non-tans are 70—80° for badan root and willow bark, 90—100° and higher for oak wood and pine bark. The percentage of tannin in total solids extracted does not depend on the temperature for oak wood and willow bark. This figure is lower in case of pine bark for temperatures above 60°, and higher for badan root at temperatures above 40°. All four materials yielded a good percentage of extract at ordinary temperatures (20—60°), viz., oak wood 57%, pine bark 59·5%, willow bark 87%, and badan root 62% of that obtained at the optimum temperature. D. WOODROFFE.

Gambier: its extraction and valuation. B. J. EATON and R. O. BISHOP (J. Soc. Leather Trades' Chem., 1926, **10**, 395—400).—A series of extracts have been prepared from the young leaf shoots and twigs of 18 months' old plants from plantations, and also from wild plants by boiling 5 lb. of each sample with 4 gals. of water in an open steam pan with constant stirring for 1 hr. The exhausted cuttings were removed, and the

liquor was strained and concentrated just below its b.p. It yielded a sticky, light mustard-yellow extract containing 50% of water and with acidity of aqueous solution of p_H 5·5. Leaves kept 1—3 days yielded softer extracts of more reddish colour and lower acidity (p_H 6—7). Evaporation of the extract at low temperatures is practised in Sumatra and yields gambier of high quality and good colour. Microscopical examination showed no vegetable *débris*, no amorphous aggregates or starch, but mostly minute needle-shaped crystals, which on melting and cooling deposit typical crystals of calcium oxalate. Additions of starch or china clay are easily detected. Chemical analysis of different prepared extracts showed them to be entirely soluble in water and to contain 22—70% of water. The tannin content of extracts prepared from plantation leaves was 38·38—52·1%, whilst that from wild leaves was 10·6—21·1%. One month's storage of the moist gambier extract without preservation induced mould growths, and the non-tans were diminished by 6·1—9·7%. The authors determined moisture by adding a few c.c. of alcohol to a weighed quantity of gambier in a flat-bottomed dish and heating to constant weight in a vacuum oven at 70°. Analyses of eight commercial cube gambiers and 14 bale gambiers showed the following percentage variations:—*Cube*: tans, 8·1—45·0; non-tans, 8·0—44·0; moisture, 4·5—15·54; acidity, p_H 4·6—5·8; ash, 5·96—69·9. *Bale*: tans, 27·8—45·0; non-tans, 39·25—68·9; moisture, 25·8—58·8; acidity, p_H 4·4—6·2; ash, 4·11—6·71. Two samples of gambier were heavily adulterated with mineral matter. D. Woodroffe.

Reaction between tannin and carbohydrates. II. H. B. Stocks and C. V. Greenwood (J. Soc. Leather Trades' Chem., 1926, **10**, 404—409; cf., B., 1925, 818).—The absorption of tannin from a concentrated solution by gum tragasol is much higher than that of gelatin, following the same order as less-concentrated solutions described (*loc. cit.*). The longer the gum tragasol and tannin solution are in contact, the greater the amount of tannin absorbed, varying from 26·3 to 74% for 6 hrs. to 6 days respectively. In the reaction between tannin and gelatin when the proportion of tannin to gelatin is 2 : 1 or 3 : 1, the composition of the washed coagulum is practically constant; tannin 66, gelatin 34. With a lower proportion of tannin to gelatin precipitation is incomplete, and the ratio of tannin to gelatin in the coagulum is less than 2 : 1. Dilution decreases the weight of coagulum obtained in the precipitation of gelatin with tannin; starch is precipitated by tannin, the amount of tannin absorbed and the weight of the coagulum being less than that obtained with gelatin or gum tragasol. The amount of tannin absorbed by starch films from solutions of various strengths is slightly higher than that of gelatin, but much lower than that of gum tragasol.

D. Woodroffe.

Reaction between tannin and casein. H. B. Stocks (J. Soc. Leather Trades' Chem., 1926, **10**, 409—410).—Alkaline solutions of two different samples of casein, an "acid" and a "rennet" casein, were respectively treated with solutions of tannin, mixed, and sufficient dilute hydrochloric acid added to render the "rennet" casein faintly acid and the other neutral. The coagulum was dried and corresponded to 255% and 261% on the original weight of the casein, which is higher than that obtained by treating gelatin with tannin. Experiments with casein and tannin solutions of different strengths showed that the casein absorbs about the same amount of tannin as does starch, but more than gelatin. D. Woodroffe.

Mangroves in Indo-China. A. H. de Balsac and A. Deforge (J. Soc. Leather Trades' Chem., 1926, **10**, 418—423).—The principal species are *Rhizophera Mucronata*, *R. Conjugata*, *R. Mangle*, *R. capsularis*, and *R. cochinchinensis*. The trees are fairly rich in tannin (10—24%), but not sufficiently so to be exported. The ratio tans : non-tans was less than 1 for two samples of *R. Mucronata*, but 2—3 for the others. Except this species, the mangroves of Indo-China should be highly suitable for extract manufacture. The colour of leather tanned with these mangroves is bad as with other mangrove-tannages, but *Ceriops* sp. ("Cay-da") is very satisfactory from this point of view. The Indo-China mangroves are, however, much inferior to those of Madagascar or the Malays. D. Woodroffe.

Water-resistant animal glue. F. L. Browne and C. E. Hrubesky (Ind. Eng. Chem., 1927, **19**, 215—219).—Animal glue 100 pts. dissolved in 225—250 pts. of water was mixed with small amounts of formaldehyde polymerides, hydrolysable formaldehyde compounds, or formaldehyde adsorption complexes, and the products gave water-resistant glued joints. The reaction of the formaldehyde with the glue was retarded sufficiently to give a working life practicable for woodworking, but the latter was extended by adding small amounts of acids. Some additions diminish the water resistance, but oxalic acid is an exception. A mixture of 10 pts. of paraformaldehyde and 5·5 pts. of oxalic acid is recommended per 100 pts. of glue in 225 pts. of water. This has a working life of 7—9 hrs. at 40—45°, and its water resistance develops slowly, attaining a maximum after 7 days. D. Woodroffe.

Gelatin viscosity and related problems. M. Briefer and J. H. Cohen (Ind. Eng. Chem., 1927, **19**, 252—257).—A gelatin gel probably consists of an elastic fibrillar structure, the individual fibres of which are swollen by imbibition until they cohere, forming as a whole an elastic gel or multiple effect of the individual fibrils. When heat is applied the fibres shrink, excreting the imbibed water of swelling. As the temperature rises, the threads begin to curl up and separate, and with further increase in temperature contract into tighter and tighter spirals. They retain their elasticity and ability to resume their original form on cooling. When the temperature and time are sufficient the spherical form of the fragments may become fixed, the elastic limit of the threads exceeded, and permanent deformation result. The viscosity of a 3% gelatin solution at 24° increases, due to the setting phase, the rapid diminution of free space outside the solid gelatin particles, due to swelling. The gelatin can be swollen and shrunk and still have its original viscosity because the number of gelatin particles present in any unit volume remains constant. The imbibition alone

varies and depends upon the relative volume or concentration of the gelatin in water. Gelatin solutions exhibit three phases, the jelly phase, the equilibrium or true solution phase, and the melting phase. Viscosity increases with time in the setting or jelly phase, but does not change in the equilibrium phase, and diminishes in the melting phase. Moderate agitation of a gelatin solution delays jelly formation, but does not appreciably affect the viscosity. Gelatins nearly alike in viscosity are not differentiated by measurements at 6·66% concentration and at 60°. At 60° the fluidity of gelatin solutions is too great to indicate their actual differences in viscosity. Wider differences are obtained with 10% solutions or with a temperature of 40—45°. Low-grade gelatins are not liable to degrade so quickly at this lower temperature. D. WOODROFFE.

PATENTS

Dyeing of leather (Swiss P. 114,271).—See IV.

Brown dye for leather (G.P. 431,776).—See IV.

Dyeing leather (G.P. 433,154).—See VI.

XVI.—AGRICULTURE.

Organic matter in heavy alkaline soils. A. F. JOSEPH and B. W. WHITFEILD (J. Agric. Sci., 1927, **17**, 1—11).—Sudan (Gezira) soils generally contain less than 1% of organic carbon. The humus carbon is about 40% of the total carbon. Alkaline humus solutions for use in the colorimetric method of determining humus keep fairly well in darkness, or in sunlight when air is excluded. Good soils have generally a higher humus content than poor soils, and there is an inverse relationship between salt content and humus content. In a vertical section, however, the maximum salt content and maximum humus content occur at about the same depth. The total nitrogen content is usually about 0·03%, of which about one-fifth is humus nitrogen. The carbon–nitrogen ratio is similar to that for English soils, namely, about 12 : 1. G. W. ROBINSON.

Moisture equivalent of heavy soils. II. A. F. JOSEPH (J. Agric. Sci., 1927, **17**, 12—20).—The moisture equivalent (Briggs–McLeane) and the imbibitional water content of purified clay fraction vary with chemical composition, method of preparation, if centrifuged, and the character of the replaceable bases. Additive relationships can only be obtained for soils of the same nature. Silt fractions show marked differences in chemical composition and moisture equivalent.
G. W. ROBINSON.

Proteins of green forage plants. III. Proteins of cruciferous forage plants (genus *Brassica*). Comparison with colzalin, a globulin from rapeseed. W. L. DAVIES (J. Agric. Sci., 1927, **17**, 33—40).—The coagulable protein from different species of *Brassica* shows practically identical distribution of nitrogen among the different amino-acids, and is closely similar in this respect to colzalin, the globulin from rapeseed. It differs in showing a lower proportion of amide nitrogen.
G. W. ROBINSON.

Proteins of green forage plants. IV. Proteins of some plants of the natural order *Umbelliferæ*. W. L. DAVIES (J. Agric. Sci., 1927, **17**, 41—43; A., 1926, 761).—Data are given for the nitrogen distribution in the proteins of parsnips and carrots. Whilst a general similarity is shown, the carrot protein contains a larger proportion of monoamino-acids, whereas in the parsnip protein there is a larger proportion of proline and hydroxyproline. The carrot protein has a higher percentage of arginine and a lower percentage of histidine than the parsnip protein, but the sum of these two amino-acids is approximately the same.
G. W. ROBINSON.

Solubility of some likely spray substances in solvents containing soap. Preparation of spraying emulsions. R. M. WOODMAN (J. Agric. Sci., 1927, **17**, 44—59).—Commercial paraffin oil is unsuitable as an insecticide owing to its low proportion of fractions boiling above 250°. The approximate solubilities of various organic liquids used in sprays, in solvents such as soap solutions, with and without addition of phenols, hydrogenated phenols, and pyridine, have been determined. Spray fluids containing paraffin oil, benzene, and aniline in solution may be economically employed, but coal-tar fractions such as anthracene and creosote oils must be applied as emulsions. Cresylic acid is the best aid to solution of paraffin oil, but is more injurious than the hydrogenated phenol, "hexalin" (*cyclo*hexanol). Emulsification in soap solutions is facilitated by rise in temperature, but the effect is more complex in the case of gelatin solutions. G. W. ROBINSON.

Losses of added phosphate by leaching from North Welsh soils. G. W. ROBINSON and J. O. JONES (J. Agric. Sci., 1927, **17**, 94—103).—Under the very humid conditions obtaining in North Wales, the effect of basic slag applied to poor pastures disappears after 6—10 years. It is shown from profile analyses that this is due to a leaching out of added phosphate from the surface layers, whereby soils tend to revert to their original phosphorus status. G. W. ROBINSON.

Action of hydrogen peroxide on farmyard manure in different stages of decomposition. G. H. G. JONES (J. Agric. Sci., 1927, **17**, 104—108).—The degree of "humification" of samples of farmyard manure of differing age was determined by the peroxide method proposed by Robinson and Jones (cf. B., 1925, 140) for soil organic matter. A good correlation was observed between the degree of decomposition of the manure as judged by its appearance and history and the degree of humification found by the peroxide method. The degree of humification varied from 35% in the case of samples containing much litter to 75% in the case of old, well-rotted samples. G. W. ROBINSON.

Studies on *Bacillus amylobacter*, A.M. et Bredemann. A. CUNNINGHAM and H. JENKINS (J. Agric. Sci., 1927, **17**, 109—117).—An aerobic coccus which appears to be a stage in the life cycle of the motile butyric acid bacillus is described. The coccus does not fix nitrogen in soil extract containing dextrose. G. W. ROBINSON.

Types of soil acidity and adsorption capacity of soils and the importance of these factors for liming and manuring with phosphorite. D. L. ASKINASI (Z. Pflanz. Düng., 1927, **A8**, 194—221).—Based on the study of a number of acid soils of different types, a classification of soil acidity is proposed, which differs somewhat from that of Kappen. Forms of soil acidity

are divided into two groups—"active" (A) and "passive" (B). Group (A) consists of acidity due to the free hydrogen ions of acids or acid salts, which can be determined directly in a water extract of the soil; group (B) includes acidity due to hydrogen ions which are adsorbed and inactive, and are detected by treatment of the soil with salts. According to the behaviour of the adsorptively-bound hydrogen ions towards salts, the latter group is subdivided into (*a*) "acidity of unsaturation," in which the hydrogen ions are displaceable by neutral salts, and (*b*) "hydrolytic acidity," in which the hydrogen is only displaced by alkalis or alkaline salts (*e.g.*, sodium acetate). The various methods proposed for determination of the acidity or "lime-requirement" of soils do not give uniform results because they determine different forms of acidity. The results of methods depending on extraction with neutral salts cannot be calculated directly in terms of the "lime-requirement" of the soil, the interaction of calcium oxide and carbonate with the soil being more complicated than the action of neutral salts. By treatment of a soil with baryta and subsequent washing with distilled water, the original adsorption capacity of the soil is increased 2—3 times. The effects on the degree of dispersion of soil colloids of saturation of the soil with sodium, with and without previous treatment with baryta, are also studied. C. T. Gimingham.

Influence of external factors on soil structure. F. Giesecke (Z. Pflanz. Düng., 1927, A8, 222—247).—Laboratory and field experiments are recorded on the effect of different types of cultivation (ploughing, harrowing, etc.) on certain properties of soils. Both soil and subsoil samples were examined. With an almost pure sand, cultivation caused no changes. With other soils, the water capacity was definitely increased, and, in most cases, there was a slightly increased power of nitrification. Wiegner's continuous sedimentation method of mechanical analysis gave good indications of the changes in physical structure brought about by cultivation. Differences between the effects due to different methods of cultivation were completely obliterated by climatic influences, particularly rainfall. Hygroscopicity and water capacity were not altered by the action of frost. It is emphasised that the methods available for this kind of investigation serve only to assist in the determination of soil structure, and do not give the real magnitude of the changes brought about by cultivation. C. T. Gimingham.

Calcium salts of phenols. Greenbaum.—See III.

Patents.

Material for control of *Peronospora*. J. H. Horst (G.P. 427,075, 12.8.21).—The material consists of a copper-lime powder, kieselguhr, casein or sodium sulphite, and sodium silicofluoride. *E.g.*, copper sulphate and sodium silicofluoride are treated with quicklime until the reaction is alkaline, and the mixture is dried and finely powdered. Kieselguhr is added, the whole mass sieved, and mixed with finely-powdered casein or sodium sulphite. A very light, bulky dust is obtained, which adheres well to foliage, and is not easily washed off by rain. C. T. Gimingham.

Material for control of *Peronospora*, mildews, and vine moths. J. H. Horst (F.P. 602,113, 14.8.25. Conv., 15.8.24. Addn. to G.P. 427,075; cf. preceding abstract).—Modification of the order in which the constituents of the dust are mixed, and addition of sodium bicarbonate and starch increase its efficiency and adhesiveness. The sodium bicarbonate prevents too rapid drying of the deposit. *E.g.*, copper sulphate is ground with kieselguhr, water added, and the whole intimately mixed with milk of lime. The product is dried, again powdered, and sieved, and is then mixed with sodium bicarbonate and starch, and repowdered. The dust thus prepared is not washed off foliage by rain, even after 3—4 weeks. C. T. Gimingham.

Process for increasing adhesiveness of dusting preparations for control of animal and plant pests. I. G. Farbenind. A.-G., Assess. of W. Bonrath, R. Lieske, and A. Thauss (G.P. 433,556, 4.4.25).—Addition of small quantities of certain compounds of cellulose with alkali and halogen-substituted aliphatic acids, prepared according to G.P. 332,203 (cf. B., 1921, 344 A), to insecticidal and fungicidal dusts, such as calcium arsenate and insoluble copper compounds, causes the dusts to adhere to foliage so that they are not removed by heavy rain. C. T. Gimingham.

Production of mono- and di-ammonium phosphate. I. G. Farbenind. A.-G., Assess. of R. Griessbach and K. Röhre (G.P. 433,742, 24.3.25).—Gases containing phosphorus pentoxide are absorbed in a solution of ammonium phosphate and the appropriate amount of ammonia is added before, during, or after absorption. C. T. Gimingham.

Means for combating plant pests. I. G. Farbenind. A.-G., Assecs. of A. Steindorff and H. Meyer (G.P. 433,653, 9.12.22).—Emulsions for spraying plants contain ammonium naphthenate, which is less harmful to the plants than naphthenic acids or their alkali or alkaline-earth salts (cf. G.P. 407,576; B., 1925, 471), together with hydrocarbons, their halogen substitution products, basic compounds, or aromatic nitro-compounds. *E.g.*, an emulsion containing ammonium naphthenate and solvent naphtha is suitable for spraying plants attacked by the aphis or caterpillars. L. A. Coles.

Fungicide. I. H. H. Jacobsen (U.S.P. 1,617,255, 8.2.27. Appl., 15.4.26. Conv., 31.7.25).—See E.P. 259,803; B., 1926, 1025.

Colloidal materials [sprays] (E.P. 263,670).—See I.

XVII.—SUGARS; STARCHES; GUMS.

Industrial production of lævulose. B. Hoche (Z. Ver. deut. Zuckerind., 1926, 821—833).—Referring to the work of the Bureau of Standards, Washington, it is remarked that the Salus-Werke in 1920—22 extracted inulin from chicory, using the methods of the beet factory, viz., diffusion, carbonation, filtration, and crystallisation, after which the inulin was hydrolysed, and the "molasses" used for the preparation of "coffee extract" or cattle food. It is admitted that the methods of the American chemists hardly appears to present any difficulty in practice, and in fact promises success. J. P. Ogilvie.

Volume of the mark in the digestion method. O. SPENGLER and C. BRENDEL (Z. Ver. deut. Zuckerind., 1926, 880—886).—Re-determinations of the volume of beet mark or insoluble matter, taking into account the so-called "colloid water," lead to the conclusion that in the hot water digestion method of determining sugar in beets the flask should be calibrated at 202·1 c.c., instead of 201·2. With a sugar content of 16·0% by the present method, the results would be 15·88% if this new correction were adopted. J. P. OGILVIE.

Determination of sugar in carbonatation scums. E. KUNZ (Z. Ver. deut. Zuckerind., 1926, 834—879).—Making an allowance for the possible effect of calcium acetate on the rotation, a number of determinations have again been made (B., 1925, 155) comparing Vondrák's method with the German official procedure, but the former is still found to be higher. The author's theory of the presence in scums of "carbonate sugar," which is decomposed under the conditions of Vondrák's method is maintained. J. P. OGILVIE.

Origin and migration of the sugars in the beet-root. O. SPENGLER and R. WEIDENHAGEN (Z. Ver. deut. Zuckerind., 1926, 767—779).—Periodical examinations of the content of various sugars present in the plant during its period of growth show that the stalks throughout contain a considerable amount of reducing sugars, dextrose predominating, and an inconsiderable amount of sucrose. In the top, sucrose is present in excess, though the dextrose content is always distinctly higher there than in the body of the root. It appears likely that the migration of sugars from leaf to root takes place in the form of monosaccharides. J. P. OGILVIE.

Purification of juice in the cane sugar industry. H. I. WATERMAN (Chem. Weekblad, 1927, **24**, 66—73).—The molasses produced in Java is found to contain a mixture of non-fermentable reducing sugars, conveniently described as glutose. This material is present to some extent in the expressed juice; it is not formed during sucrose extraction from the glucose present, but from the fructose, or other reducing sugars sensitive to alkalis. High alkalinity and slow working encourage the formation, but evaporation and heating do not affect it. Molasses from Java and Egypt contained 6%, from Cuba nearly 10%. It is important, therefore, to keep the concentration of lime as low as possible during the manipulation of the juice, and the methods of purifying the solutions are considered from this aspect. The double carbonatation method of de Haan, with subsequent sulphitation, is considered the most suitable for this purpose. S. I. LEVY.

Destruction of sugar during the evaporation of juice [in beet factories] using high temperatures. P. ORTH (Bull. Assoc. Chim. Sucr., 1926, **43**, 458—488, **44**, 5—12).—Using an experimental evaporator with high-temperature steam, and operating so as always to maintain the dry substance at a constant value, a large number of tests were conducted with second-carbonation juice and with diluted syrup. It was, however, not found possible to obtain any accurate values for the destruction of sucrose, owing to the formation of dextrorotatory substances, which vitiated the double-polarisation method used for the determination of the sucrose. On considering only the minimum values obtained for the destruction of sugar, the general conclusion arrived at is that the use of temperatures greater than 115° is uneconomical in evaporation, the value of the sugar then lost exceeding that of the coal saved in the high-temperature evaporation. J. P. OGILVIE.

PATENTS.

Refining unwashed sugar. Refining sugar with "Carbrox." C. J. GAMBEL (U.S.P. 1,606,215—6, 9.11.26. Appl., 13.5.22).—(A) A solution of unwashed raw sugar is clarified and partially decolorised by treatment with a mixture of lime, an inert filtering material, and a decolorising carbon ("Carbrox"), the solution being maintained alkaline. This is filtered and the filtrate finally decolorised and concentrated to produce a refined sugar. (B) A solution of sugar is clarified and filtered, and the filtrate partially decolorised in an agitator by the action of a quantity of "Carbrox" equal to 0·75% of the sugar present. This solution is again filtered and concentrated. J. P. OGILVIE.

Manufacture of crude lactose. R. W. BELL, Assr. to GOVT. AND PEOPLE OF U.S.A. (U.S.P. 1,600,573, 21.9.26. Appl., 15.4.26).—Casein and fat being removed, the whey is adjusted to p_H 7·0 (approx.) by adding a suitable alkali, after which it is heated at about 60° and concentrated at a temperature below the coagulating point of the albumin contained therein to a density at which the lactose just begins to crystallise, the temperature being subsequently lowered to 0° and there maintained until the maximum crystallisation has occurred. J. P. OGILVIE.

Production of crystalline dextrose from starch-bearing materials. CORN PRODUCTS REFINING CO., Assees. of W. B. NEWKIRK (E.P. 246,098, 11.8.25. Conv., 17.1.25).—A preliminary crystallisation is induced, the solid dextrose thus obtained being partially melted, so as to leave some of the crystals in the liquid, and the magma subjected to a final crystallisation while maintaining the solid phase dispersed through the liquid. The solid dextrose of the preliminary crystallisation may be either anhydrous or hydrate, and the final crystallisation is carried on at temperatures favourable to the type of crystals predominating in this first crystal mass. The mass of first crystals is then centrifuged. J. P. OGILVIE.

Production of tricalcium saccharate rich in sugar and poor in lime and of a very high degree of purity [from beet molasses]. C. STEFFEN (E.P. 252,110, 1.8.25. Conv., 18.5.25).—Tricalcium saccharate is obtained by mashing saccharate obtained from a molasses solution into a cold low-percentage sugar solution, 1½—3 times the quantity of sugar in the sugar solution being added in the form of saccharate, and then adding lime powder to precipitate the calcium saccharate dissolved in the mixture, the saccharate being finally separated, *e.g.*, by filter-pressing. J. P. OGILVIE.

Electrolytic reduction of sugars to alcohol. H. J. CREIGHTON, Assr. to ATLAS POWDER CO. (U.S.P. 1,612,361, 28.12.26. Appl., 26.3.26).—Reduction of sugars to alcohols, *e.g.*, mannose to mannitol, is carried out in an electrolytic cell having an anode coated with lead dioxide

and surrounded by a porous diaphragm, and a cathode consisting of a layer of mercury at the bottom which can be mechanically stirred. The anode compartment contains 5% sodium sulphate solution, and the cathode compartment a mixture of that electrolyte with sugar solution (15—25%). On passage of the current (1—1½ amp./dm.^{2} and 5—8 volts), the sugar is reduced with formation of sodium hydroxide, while oxygen is liberated in the anode compartment, the solution becoming acid with sulphuric acid. The diaphragm, while preventing the diffusion of the sugar and alcohol solution into the anode compartment, is sufficiently porous to allow the migration of hydrogen and hydroxyl ions, so that there is a continuous re-formation of sodium sulphate in the two compartments. The solution in the cathode compartment remains neutral or very slightly alkaline, a condition which is favourable to the reduction of sugars by nascent hydrogen. F. R. Ennos.

XVIII.—FERMENTATION INDUSTRIES.

Scientific control in the preparation of bottled champagne. E. Manceau (Ann. Falsif., 1927, 20, 7—19).—Control of the preparation of champagne comprises a rapid analysis of the "projet de cuvée" consisting of a determination of density, alcohol, total acidity, and sugar, and sometimes sulphurous acid, and a microscopical examination. The proportion and nature of the wines to be mixed to form the "cuvée de tirage" is fixed by taste and on the mixture are determined density, alcohol, total and volatile acidity, sugar, dry extract, ash, potassium tartrate and free tartaric acid, tannin, total and ammoniacal nitrogen, and total and free sulphurous acid. From these results are determined the weight of tannin and of fining to be added, and the first evaluation of sugar to be added is made. After 2—4 weeks the clear wine is examined as before, and the proportion of sugar that it should contain when bottled determined, and the analysis repeated after addition of the "liqueur de tirage." The same determinations are again made after the second fermentation, and in addition the pressure at 0° is found and a microscopical examination of the deposit often made. A final analysis is made after the shaking and clearing of the wine. Details are given of the methods of analysis used, and variations in composition discussed. Scientific control has reduced the usual loss by explosion of bottles, made unsuccessful fermentation exceptional, and defined conditions resulting in "maladie du Bleu" which affects clarity. D. G. Hewer.

***Bac. amylobacter*, A.M. et Bredemann.** Cunningham and Jenkins.—See XVI.

Patents.

Simultaneous dehydration and purifying of alcohol. Distilleries des Deux-Sèvres (E.P. 253,128, 3.6.26. Conv., 4.6.25).—In the azeotropic distillation of crude alcohol in apparatus similar to that described in E.P. 243,368 (cf. B., 1926, 604), esters are hydrolysed and acids are neutralised by the introduction of concentrated sodium hydroxide solution into the column at a point below that at which the first distilled products are eliminated. When esters are not present, neutralisation of the acids is not essential. High-boiling impurities, such as alcohols, aldehydes, bases, esters, and acids, are concentrated in the column below the point at which the anhydrous alcohol is withdrawn, the lower end of the column being extended, if necessary, and are subsequently rectified in an auxiliary column which returns any alcohol to the main column. L. A. Coles.

Process of making proteolytic enzymes. S. A. Waksman (U.S.P. 1,611,700, 21.12.26. Appl., 19.1.20).—A suitable culture is propagated and the successive generations resown on a medium containing protein. The spores are then developed for several days at 30° on a sterilised mixture of wheat, bran, bean cake, and alfalfa meal with water, whereby the proteolytic properties are greatly increased. The resulting mass or the separated spores are further developed on a similar medium for 24—48 hrs. at 28—32°. The product is either dried, ground, and used as such, or the enzymes may be obtained from the water extract by concentration or precipitation with alcohol or ammonium sulphate. F. R. Ennos.

Alcohol from sugar (U.S.P. 1,612,361).—See XVII.

XIX.—FOODS.

Composition of milk. I. Variation in solids-not-fat, fat, and protein of cow's milk, and their relationship. H. T. Cranfield, (Miss) D. G. Griffiths, and E. R. Ling (J. Agric. Sci., 1927, **17**, 62—71).—The percentages of fat, solids-not-fat, and protein respectively for 700 samples of mixed milk taken from 15 different herds during 1925—6 are reported. Frequency distributions are shown and standard deviations calculated for each constituent. In the curve showing the correlation between fat and solids-not-fat, the percentage of fat appears to pass through a minimum which corresponds with the average percentage (8·8) of solids-not-fat. There is a marked correlation between protein and solids-not-fat. There is no definite seasonal effect on protein percentage, but there is a tendency for the ratio solids-not-fat : protein to be low in summer and high in winter, so that deficiency in solids-not-fat would appear to be due to lactose deficiency in summer and to protein deficiency in winter. G. W. Robinson.

Composition of milk. II. Variation in percentage of mineral constituents in cow's milk, and their relationship with solids-not-fat and protein content. H. T. Cranfield, (Miss) D. G. Griffiths, and E. R. Ling (J. Agric. Sci., 1927, **17**, 72—93; cf. preceding abstract).—Ash data are given for 670 samples of mixed milk from 15 different herds. The correlations of the different constituents are shown by means of graphs. Total ash falls with solids-not-fat until, when low values of the latter constituent are reached, the ash content tends to rise. This was also shown in studies of the milk of individual cows. Soluble ash varies inversely and insoluble ash directly with solids-not-fat. Lime and phosphoric acid vary with solids-not-fat. Total ash and soluble ash reach a maximum in summer, whilst insoluble ash shows a minimum in May and a maximum in autumn and winter. Phosphoric acid shows little seasonal change, but lime falls in spring and summer, and rises in autumn. G. W. Robinson.

Evaluation of sour cream. M. A. Dichno and O. M. Briskin (Z. Unters. Lebensm., 1926, **52**, 469—475).—To be classed as sour, a cream sample should be character-

ised by the following:—d 1·0135; fat, not less than 23·46%; acidity, not less than 0·7% of lactic acid; coloration in reductase test after 2 hrs.; clotting in fermentation test after 2½ hrs. The importance of care in the commercial handling of milk is emphasised.

A. G. POLLARD.

Loss of mineral matter from potatoes during ordinary culinary preparation. C. GRIEBEL and A. MIERMEISTER (Z. Unters. Lebensm., 1926, **52**, 458—461).—Cooking water from boiling potatoes contains considerable amounts of calcium, magnesium, and potassium salts, chlorides, sulphates, and phosphates. Unpeeled potatoes lost 5·8% of their mineral content on boiling in water, and only 1·4% when steamed. Peeled potatoes lost 17% of minerals on boiling and 7·1% when steamed. The manner of cooking (steaming or boiling) considerably affected the relative proportions of the minerals appearing in the cooking water. Boiling in salt water considerably reduced the loss of potassium salts.

A. G. POLLARD.

Determination of milk fat in foodstuffs by the Reichert–Meissl value. H. LÜHRIG (Pharm. Zentr., 1927, **68**, 49—54, 65—68).—In the determination of milk fat in foodstuffs, containing other fats such as coconut oil, palm kernel oil, etc., by means of the table of Kuhlmann and Grossfeld (B., 1926, 252), it is not always possible to obtain 5 g. of fat for the Reichert–Meissl value. The original semi-microchemical determination (B., 1922, 508 A) is not satisfactory, and for the above fat mixtures a modified microchemical method which has been found to give substantially correct results is as follows: 0·5 g. of fat and 2 g. of glycerin are saponified with 0·2 c.c. of sodium hydroxide solution (1 : 1). 10 c.c. of hot water and 5 c.c. of sulphuric acid are added, and 14 c.c. are distilled over in 5 min. 10 c.c. of the filtrate are titrated with 0·025N-alkali. The titration figure multiplied by 1·4/0·4 gives the Reichert–Meissl value corresponding to 5 g. of fat.

F. R. ENNOS.

Solidification point of edible fats. MEYER. **Sorting of butters and margarines.** MANLEY. **Determination of butter fat in margarine.** ELSDON and SMITH.—See XII.

PATENTS.

Process of treating grain and product produced thereby. J. A. WESENER (E.P. 263,213, 13.10.25).—The unhusked grain, more especially oats, is treated with an aqueous solution of an alkali or alkaline earth in predetermined quantity, sufficient to neutralise the natural acids and those developed in the subsequent processes, without making the product alkaline. After cooking the grain under steam pressure, the husks are ruptured and the whole is dried; the husks are subsequently removed and the treated kernels made into the desired form.

F. R. ENNOS.

Method of producing groats from the soft endosperm of grains of cereals. L. BARTMANN (E.P. 254,748, 3.7.26. Conv., 3.7.25).—After steeping a cereal grain, the endosperm is expelled from the husks in a pasty condition and moulded into groats by forcing it into metal tissue or into the perforations in metal. After drying in these moulds, it is suitably removed.

F. R. ENNOS.

Manufacture of meat juice. LES ÉTABL. BYLA (F.P. 606,962, 10.3.25).—Coagulation of the proteins is prevented by macerating finely-chopped meat with water containing papain at about 46°. L. A. COLES.

Method and apparatus for the manufacture of food for dogs and other animals. MOLASSINE CO. and H. C. DAVIS (E.P. 264,278, 13.11.25).—Sterilised meat and finely divided dog biscuit in a horizontal, steam-heated cylinder are subjected to jets of steam, which may be superheated at low pressure. A binding material consisting of flour and water is sprayed in under air pressure, while rotating paddles mix the ingredients, causing them to pass from one end of the cylinder to the other, where they are discharged, moulded, and dried.

F. R. ENNOS.

Dessication of milk (E.P. 257,893).—See I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Salts of linolenic hexabromide from lumbang oil. G. A. IMPERIAL and A. P. WEST (Philippine J. Sci., 1926, **31**, 441—449).—Hexabromolinolenic acid, m.p. 179·5—180·5°, prepared from lumbang oil, has been used in the preparation of a number of its metallic salts. Potassium hexabromolinolenate (cf. Erdmann and Bedford, A., 1909, i, 357), obtained by direct neutralisation of the acid in hot benzyl alcohol solution, turns brown at 180°, m.p. with decomp. 185—190°. Double decomposition of this salt with barium bromide, both in hot methyl alcohol (95%) solution, gives barium hexabromolinolenate (*ibid.*) which browns at 197° and blackens without melting at 200°, whilst similar treatment with zinc chloride gives *zinc hexabromolinolenate*, decomp. sharply at 174°. *Lead hexabromolinolenate*, prepared from the free acid and lead acetate in hot ethyl acetate and alcoholic solution, respectively, browns at 160°, m.p. with decomp. 190—195°. A table is given showing the relative solubilities of the free acid and the four salts in twenty-eight different solvents, and it is specially noted that free hexabromolinolenic acid is soluble to the extent of 5% in warm xylene and in benzyl alcohol at 100°, and 1% soluble in warm *iso*butyl alcohol.

E. HOLMES.

Volumetric determination of commercial synthetic vanillin. V. ZANOTTI (Boll. Chim. Farm., 1927, **66**, 33—34).—Vanillin may be determined by titration with 0·1N-sodium hydroxide solution in presence of phenolphthalein: 1 c.c. of 0·1N-alkali = 0·0152 g. of vanillin. If the product contains 2% of acetanilide, which is the commonest impurity of commercial vanillin, rather low results are given by the titration.

T. H. POPE.

Pine-needle extracts. M. KLOSTERMANN and H. QUAST (Z. Unters. Lebensm., 1926, **52**, 476—478).—To detect sulphite-cellulose liquor in adulterated pine-extracts, the determination of sulphate in the ash is valuable. Adulterated samples contained three times the sulphate content of pure material. The percentage of magnesium, iron, aluminium, and phosphate in the ash of adulterated extract was less than in the pure extract, and the total mineral substance in the pure extract was very low.

A. G. POLLARD.

Conversion of ethylene into alcohol. GLUUD and SCHNEIDER.—See II.

PATENTS.

Production of oxygen-containing and other organic compounds. H. DREYFUS (E.P. 263,503, 1.7.25).—Methyl alcohol, formaldehyde, keten, acetic acid, and other aliphatic or cyclic oxygenated compounds, and also higher hydrocarbons, are formed from mixtures of carbon monoxide and hydrogen, or gases containing them, at about atmospheric pressure, in the presence of catalysts, other than those (such as nickel, iron, and cobalt) which favour the production of methane. As catalysts, there may be used, for the production of methyl alcohol, zinc oxide (cf. E.P. 262,494; B., 1927, 124) or the substances mentioned in E.P. 227,147 and 229,714—5 (B., 1925, 189,338), and 20,488/13 (B., 1915, 788); for acetic acid, the substances mentioned in E.P. 262,832 (B., 1927, 125); and for keten, those mentioned in E.P. 262,364 (B., 1927, 125). In all these specifications high pressures are indicated. Apart from the modification in this sense, the processes are carried out in the present instance substantially under the conditions (temperature, proportions, etc.) described in the specifications. The gases used may be freed from impurities such as organic sulphur compounds and volatile iron compounds prior to use. B. FULLMAN.

Ester of [*n*-]butyl alcohol. R. H. VAN SCHAACK, JUN. (U.S.P. 1,613,366, 4.1.27. Appl., 21.8.25).—Esters of *n*-butyl alcohol with saturated aliphatic acids containing more than 8 carbon atoms in the molecule, *e.g.*, n-*butyl stearate*, m.p. 18°, render nitrocellulose films soft and flexible, and are of value in the manufacture of lacquers. T. S. WHEELER.

Manufacture of esters of oxy[keto]-acids [pyruvic acid]. C. H. BOEHRINGER & SOHN, Assees. of A. HAUSSLER (U.S.P. 1,614,195, 11.1.27. Appl., 20.2.25. Conv. 5.3.23).—An ester of a hydroxy-acid, *e.g.*, ethyl lactate, is passed in the vapour state with air or other gas containing oxygen or ozone over a catalyst, *e.g.*, vanadium pentoxide or molybdenum trioxide, at 100—500°, when the corresponding keto-acid ester, *e.g.*, ethyl pyruvate, is obtained in a yield of about 60%. T. S. WHEELER.

Production of esters of volatile organic acids. I. G. FARBENIND. A.-G., Assees. of G. VON BRÜNING and O. NICODEMUS (G.P. 434,279, 21.11.24).—Mixtures of alcohols and acids in the form of vapour are passed over active charcoal at raised temperatures. As the presence of water has no effect upon the yield of ester, dilute acids or alcohols can be used; *e.g.*, a 90·8% yield of ethyl formate is obtained by passing 79% formic acid and alcohol vapour over active charcoal at 150°. L. A. COLES.

Manufacture of a liquid chlorothymol preparation. F. RASCHIG, CHEM. FABR. (G.P. 433,293, 3.2.25).—Chlorothymol is melted with camphor. *E.g.*, by melting 10 pts. of chlorothymol with 5 pts. of camphor a mixture is obtained which is liquid at all temperatures above 5°, and may be used as a dental disinfectant. E. H. SHARPLES.

Making ethylene glycol monoalkyl ethers. J. G. DAVIDSON, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,614,883, 18.1.27. Appl., 7.2.24).—Ethylene glycol (2 mols.), sodium hydroxide (2 mols.), and ethyl sulphate (1 mol.) are boiled under reflux for 3 hrs., and distilled *in vacuo* when ethylene glycol monoethyl ether is obtained in a yield of 60%. T. S. WHEELER.

Manufacture of lead alkyl compounds [lead tetraethyl]. F. W. SULLIVAN, JUN., and L. CHALKLEY, JUN., Assrs. to STANDARD OIL CO. (U.S.P. 1,611,695, 21.12.26. Appl., 6.10.24).—Sodium–lead alloy containing 10% Na (100 pts.), ethyl sulphate (50 pts.), toluene (65 pts.), potassium iodide or other alkaline or alkaline-earth halide (0·5 pt.), and a trace of water are boiled under reflux with agitation for 4—6 hrs., and the lead tetraethyl, which is formed in a yield of 80%, is recovered by distillation with steam, or *in vacuo*, or by extraction with ether. T. S. WHEELER.

Manufacture of barbituric acid derivatives. J. D. RIEDEL A.-G. (E.P. 244,122, 5.12.25. Conv., 6.12.24).—Disubstituted barbituric acids in which the substituents are an unsaturated radical (sometimes halogenated, *e.g.*, β-bromo-Δ^{β}-propenyl) and a radical of the type $-CHR_1R_2$ (R_1 and R_2 being different aliphatic, aromatic, or alicyclic radicals), show much increased soporific action, which is ascribed to the asymmetry produced in the carbon atom connecting the second substituent to the barbituric acid residue. For the introduction of the β-bromo-Δ^{β}-propenyl group into diethyl malonate or barbituric acid derivatives either βγ-dibromo-Δ^{β}-propene or αβγ-tribromopropane may be used. The following barbituric acid derivatives are described: 5-iso*butyl*-, m.p. 194—195°; 5-iso*butyl*-5-Δ^{β}-*propenyl*-, m.p. 109—110°; 5-iso*butyl*-5-β-*bromo*-Δ^{β}-*propenyl*-, m.p. 131—132°; 5-α-*methylbutyl*-, m.p. 162—163°; and 5-α-*methylbutyl*-5-β-*bromo*-Δ^{β}-*propenyl*-, m.p. 164—165°: and also *diethyl α-methylbutylmalonate*, b.p./13 mm. 122—125°. B. FULLMAN.

Methods for producing scarlet fever toxin and antitoxin. G. F., and G. H. DICK (E.P. 243,675, 31.8.25. Conv., 28.11.24).—Broth is inoculated with a pure culture of hæmolytic streptococci specific to scarlet fever, and a little blood is added. After incubation, the bacteria are removed by passage through a porcelain filter, or killed by heat treatment or treatment with an antiseptic. To prepare the antitoxin, sterile toxin is inoculated in horses, the blood-serum or blood-plasma of which is concentrated or refined by precipitation with ammonium sulphate. The latter is removed from the precipitate by dialysis, and the antitoxin dissolved in physiological salt solution with the addition of a preservative. B. FULLMAN.

Emulsifying and dissolving media. E. SCHMIDT (G.P. 432,942, 11.12.23).—These consist of the water-soluble salts of hydroaromatic (especially of polynuclear) carboxylic acids, substituted by higher aliphatic radicals such as propyl, butyl, and amyl. Thus, by adding butyltetrahydronaphthalenecarboxylic acid to tetralin, a clear solution is obtainable on dissolving in water. R. A. A. TAYLOR.

Manufacture of pyridine-3-carboxylic acid amides. M. HARTMANN and M. SEIBERTH, Assrs. to SOC. OF CHEM. IND. IN BASLE (U.S.P. 1,617,332, 15.2.27. Appl., 11.12.25. Conv., 20.12.24).—See E.P. 244,747; B., 1926, 720.

Preserving specimens (E.P. 263,674).—See I.

Preparation of 3-nitro-4-aminobenzoyl-o-benzoic acid (U.S.P. 1,614,584).—See IV.

Preparation of 2 : 5-diaminobenzene-1 : 4-dicarboxylic acid (G.P. 433,277).—See IV.

Polymerised styrene (U.S.P. 1,613,673—4).—See XIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Difference between the reducing powers of metoquinone and a mixture of methyl-*p*-aminophenol sulphate [metol] and quinol. A. and L. LUMIÈRE and A. SEYEWETZ (Bull. Soc. Franç. Phot., 1926, [iii], **13**, 312—314).—Reply to Hübl (Phot. Korr., 1926, **62**, 1).—It is maintained that the result of mixing in solution 2 mols. of metol and one of quinol is a stable compound rather than an addition compound which dissociates in solution. An analogous example of salt formation between developers possessing acid and those possessing basic groups is provided by hydramine (*p*-phenylenediamine + quinol). W. CLARK.

Desensitisation. A. P. RICHARD (Bull. Soc. Franç. Phot., 1926, [iii], **13**, 331—332).—Desensitisation of a panchromatic plate was studied, using the chief commercial desensitisers, by desensitising and exposing under a wedge provided with blue, green, and red filters. The results obtained are tabulated. Some emulsions readily fog in presence of desensitisers, but it is always possible to find a desensitiser which does not fog a given emulsion, provided a suitable developer is used. W. CLARK.

Hypersensitisation. R. MAUGE and A. P. RICHARD (Bull. Soc. Franç. Phot., 1926, [iii], **13**, 332—335).—A 3% ammonia solution with no silver salts gave slightly less fog than the Ninck hypersensitiser containing silver salts, for a sensitivity of two and a half times less; slightly superior results were obtained with a 2% solution. Ninck's formula is superior from the point of view of rapidity, and is preferable. With autochrome plates, a slight intensification is necessary to give the brilliance and depth of colour desired. Plates treated with pantochrome are less contrasty than those treated with pinachrome–pinacyanol. The rendering of autochromes can be improved by colour sensitising, in which case, for use with over-run electric lamps, a colour screen is not needed. In open air, the screen recommended by Ninck is suitable. Other aspects of hypersensitisation and desensitisation are considered. With illumination over-run 20% in voltage, the amount of pinacyanol can be reduced by one-third, owing to the higher red content of the light, compared with daylight. W. CLARK.

PATENTS.

Colour kinematography. TECHNICOLOR MOTION PICTURE CORP. (E.P. 264,369, 12.4.26).—Means are described for making complementary kinematograph films preparatory to dyeing the films different colours for use as matrices in mechanical printing processes or to cementing the two films in super-position to form a composite film in natural colours. The general principle is dealt with, and a mechanical arrangement is described in detail. W. CLARK.

Photographic process. F. MÜHLING (F.P. 607,915, 12.12.25).—The coated emulsion layer has a maximum thickness of 0·005 mm. and contains only the amount of silver required for the production of the image. Development is very rapid, *e.g.*, 1·5 min. W. CLARK.

Applying photographic baryta or emulsion coating. MIMOSA A.-G. (G.P. 433,387, 29.1.26).—The material is applied to the support with the aid of reduced pressure. Separate parts of the supports can also be coated. W. CLARK.

Developer for photographic plates, films, papers, and the like. K. BINDER (U.S.P. 1,617,434, 15.2.27. Appl., 25.8.25. Conv., 14.7.25).—See F.P. 600,801 ; B., 1926, 566.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Low-density dynamite. J. MARSHALL, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,614,447, 11.1.27. Appl., 30.7.24 ; cf. 1,113,275 and 1,124,679 ; B., 1914, 1116 ; 1915, 251).—Dried ground maize stalks, when used as an absorbent in the preparation of ammonium nitrate dynamites, give a product which has a low density and a low velocity of explosion, and is suitable for use as a safety explosive in mines. T. S. WHEELER.

XXIII.—SANITATION; WATER PURIFICATION.

Volumetric determination of sulphate in drinking water. A. BAHRDT (Z. anal. Chem., 1927, **70**, 109—119). —To remove the metallic ions in the water a small quantity of sodium hydrogen carbonate is added (to neutralise any acidity) and 300 c.c. are filtered through 10 g. of washed sodium permutit contained in a small bulb tube. The filtrate contains only alkali metals together with all the sulphate ions ; 200 c.c. are neutralised with 0·1*N*-hydrochloric acid using methyl orange as indicator and 1 c.c. excess of acid is added. After boiling to expel carbon dioxide, 10—30 c.c. of 0·1*N*-barium chloride solution are added to precipitate the sulphate, an aliquot part of the filtrate is neutralised exactly with sodium hydroxide, and the excess barium chloride titrated with 0·1*N*-potassium palmitate using phenolphthalein as indicator. The palmitate solution is standardised against the barium chloride solution and is prepared by adding a 16% solution of potassium hydroxide in propyl alcohol to a warm mixture of 26 g. of palmitic acid, 500 c.c. of propyl alcohol, 250 c.c. of water, and 0·1 g. of phenolphthalein until a faint pink colour is obtained, filtering, and diluting to 1 litre. The permutit may be regenerated after use by washing with 5% sodium chloride solution then with cold water. A. R. POWELL.

Treating steam-boiler water. Treatment of boiler water. R. E. HALL, Assr. to J. M. HOPWOOD (U.S.P. 1,613,656 and 1,613,701, 11.1.27. Appl., [A] 28.6.26 ; [B] 6.6.24).—See Hall, B., 1925, 421.

Means for cleaning sand filters of large surface. A. SIVADE (U.S.P. 1,617,654, 15.2.27. Appl., 18.10.23. Conv., 20.10.22).—See E.P. 205,818 ; B., 1924, 652.

Analysis of liquids [water] (E.P. 264,237).—See I.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 15, 1927.

I.—GENERAL; PLANT; MACHINERY.

Mechanism of filtration. E. E. Jewett and R. E. Montonna (Chem. Met. Eng., 1927, 34, 86—91).—Filtration constants for calcium carbonate and calcium sulphate were determined using a 12-in. plate and frame filter press with No. 20 cotton duck as filter cloth. The equation of Almy and Lewis (cf. B., 1912, 859) $R = KA^2P^n/V^m$ was applied and the constants m, n, and K were determined under varying conditions of pressure, concentration of sludge, viscosity, and surface tension of filtrate. The value of m was 1 for each sludge, and curves given show that m is independent of the pressure of filtration up to the limit of the experiment, 22 lb./sq. in., and also that it is not affected by the concentration of solids in the sludge, nor by pre-filtering or pre-drying. The authors consider that n is also independent of these three factors, and that an average value of 0·85 for n will hold for all incompressible solids. The value of K includes the variable effects of temperature, solid concentration, viscosity, and surface tension, and a simplified equation for its calculation is evolved. Graphs show that calcium carbonate at lower concentrations filters much more readily than does calcium sulphate, but that the rate of filtration of calcium carbonate falls off more rapidly with increase of solids, *i.e.*, calcium sulphate gives a drier cake. Curves also show that the viscosity or surface tension of the filtrate produces a decrease in the rate of filtration, and the addition of alkali has the same effect.

W. G. Carey.

Recovery [of volatile solvents] by the Brégeat system. Brégeat A.-G. (Chem.-Ztg., 1927, 51, 101—103).—A disadvantage of this process for the recovery of alcohol, ether, acetone, etc. from large volumes of air is the resinification or polymerisation of the cresol used for absorption. This is not due to repeated heating in presence of air, for resinification still occurred when distillation of the solvent *in vacuo* was tried at 70° instead of 140°. In one plant investigated the air–vapour mixture contained as impurities volatile acids from the nitration of cellulose, and from the ether used, as well as vegetable and mineral dust; this last item when dealing with 30,000 cub. m. of air–vapour mixture per hr. amounted to 100 kg. in 24 hrs., which was held back by the cresol and increased its viscosity. These impurities were eliminated by filtration of the air–vapour mixture through special filtering media treated with soda solution. Naphthalene or tetralin lessens the tendency of the cresol to resinify, but a large amount of the former may block the plant, whilst excess of tetralin decreases the absorptive capacity of the cresol.

W. G. Carey.

Solid carbon dioxide as refrigerant. Killeffer.—See VII.

Patents.

Recovering adsorbed material from adsorptive material. G. Müller, Assr. to American Lurgi Corp. (U.S.P. 1,617,960, 15.2.27. Appl., 6.2.25).—The adsorptive material is placed in heat-exchanging relation with circulating hot gases. Fresh hot gas is introduced at a point in the cycle in front of the heat-exchanging zone, and a portion of the gas is withdrawn at a point behind this zone.

H. Holmes.

Method and apparatus for determining hydrogen-ion concentration. E. D. Stirlen (U.S.P. 1,616,092, 1.2.27. Appl., 8.9.23).—Two samples of the material are placed in separate receptacles, and with one sample is mixed a known quantity of a suitable reagent. A beam of light is passed through the mixture, and a second beam is passed through the pure sample and through a double wedge prism to produce a similar colour in the emergent beam.

H. Holmes.

Furnaces. E. J. Vermeyen (E.P. 239,206, 25.8.25. Conv., 29.8.24).—Coal is distilled in an upper chamber, and the residue falls into a lower or fire chamber, the heat from which effects the distillation. The upper chamber may be formed as an arch over which the coal is conveyed by a scraper conveyor, or as a rotary cylinder.

B. M. Venables.

[Rotary] furnaces and the like. G. Martin and J. D. Taylor (E.P. 264,919—20, 27.10.25).—(a) A rotary kiln is constructed of a metallic skeleton surrounded by material of low conductivity, such as cement. (b) A rotary kiln or dryer is provided with a number of transverse semi-circular baffles to cause the gases to take a sinuous course and promote contact between them and the material under treatment. The baffles may be constructed of tubes or of metallic lattice-work covered with cement or like material.

B. M. Venables.

Rotary kilns and furnaces. J. H. Bentley (E.P. 265,462, 8.1.26).—A rotary kiln is provided with an extension beyond the flame or sintering zone which acts as an air preheater and product cooler. The interior is provided in the earlier part with lifters which drop the material through the air stream, and at the end with scoops which lift the material to a central outlet, from which the material drops down a stationary conduit for the incoming gas to a conveyor, the whole system being arranged for heat interchange.

B. M. Venables.

Method of heating furnaces. King, Taudevin & Gregson, Ltd., and C. Nelson (E.P. 264,938, 30.10.25).—A quantity of combustible gas and a restricted quantity of secondary air are admitted to a chamber adjoining

the combustion chamber, and pass into the latter partly at floor level and partly above the floor. Tertiary air is admitted at a still higher level. All the admission ports are on one side of the furnace, and the gases pass across the combustion chamber to exhaust flues opposite. The secondary air and, if desired, also the tertiary is preheated by heat from the gas conducted through the walls of the supply passages. B. M. VENABLES.

Method and apparatus for transferring heat. A. E. WHITE. From GAS RESEARCH Co. (E.P. 265,358, 12.1.26).—The heat from the hot fluid which is, for instance, outside the recuperator tubes is transferred to them by contact, and on arriving at the inside of the tubes is radiated to thin plates, wire mesh, or other filling having a large surface, being finally transferred to the fluid to be heated by contact with this much increased surface. B. M. VENABLES.

Apparatus for effecting heat transfer. H. F. SMITH, Assr. to GAS RESEARCH Co. (U.S.P. 1,617,609, 15.2.27. Appl., 16.3.22).—The material to be heated is placed in a chamber flared at its ends, and hot furnace gases are delivered at high velocity through a nozzle into one end. The gases, after passing through the chamber, are returned through a passage connecting the two ends and terminating around the nozzle, and thereafter are in part recirculated through the chamber and in part escape through a vent pipe. H. HOLMES.

Packings for apparatus working at high pressures. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 264,988, 24.12.25).—A single sheet of natural mica less than 0·5 mm. thick is used as a gasket between flat metallic surfaces; it is resistant to high temperatures and pressures (over 100 atm.), and is an electrical insulator. B. M. VENABLES.

Preventing the entrance of moisture into heat-insulating devices. E. SCHMIDT and E. DYCKERHOFF (E.P. 262,103, 22.11.26. Conv., 30.11.25).—To prevent the accumulation of moisture in, and consequent loss of heat-insulating property of, the lagging of refrigerating machines, the lagging is approximately enclosed in a metal casing, the passage of air to or from the interior being freely permitted under changes of temperature, but only through vents containing calcium chloride or other hygroscopic material. B. M. VENABLES.

Boiling or raising the temperature of water and other liquids. C. F. HAMMOND and W. SHACKLETON (E.P. 265,253, 5.9.25).—A burner is submerged in one liquid, *e.g.*, molten metal, and the heat is conducted from this liquid through a partition wall to the liquid to be heated. The rate of heat transference from the flame to the second liquid is increased because there is no gas film on the partition. B. M. VENABLES.

Producing a protective coating in water tubes for the purpose of preventing incrustations. C. BÜCHER (E.P. 260,233, 5.10.26. Conv., 21.10.25).—Sufficient calcium hydroxide solution is added to a drinking-water supply containing free or partly bound carbonic acid to produce calcium carbonate. This forms over a period of months a protective chalk lining in the iron pipes, while the hydrogen-ion concentration is increased above the neutrality point without exceeding the palatable limit. H. HOLMES.

Means and method for preventing and removing scale and incrustation in steam boilers. G. S. NEELEY and G. WATKINS (E.P. 264,551, 8.9.25).—An alternating current is applied to the boiler at points of maximum temperature difference. Preferably one lead is connected to the lower part of the boiler, and the other lead, insulated from the boiler wall, is connected to an electrode suspended immediately beneath the surface of the water. [Reference is directed under Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 147,528 (B., 1921, 455 A), 6105/92, and 3749/87.] H. HOLMES.

Drying machines. STOTHERT & PITT, LTD., and W. MINTY (E.P. 264,921—2 and 264,931, [A and B] 27.10.25; [C] 29.10.25).—A rotary drying drum for materials such as road metal is provided in (A) with a charging chute, which swings out of the heat and forms a door when not in use; in (B) with internal lifters of such a shape that the larger pieces of material are deflected backwards and delayed in the dryer; and in (C) with an end bearing so situated as to remain cool. B. M. VENABLES.

[Combined machine for] drying and grinding wet materials. F. W. DAVIS (E.P. 265,070, 31.7.26).—A rotary drum is divided into three compartments, in all of which drying is effected by hot gases passing through, and in two of them, either the first and third or second and third, grinding is also effected. B. M. VENABLES.

Mixing apparatus. S. W. M. WHITEHEAD (E.P. 265,382, 11.2.26).—A proportioning device combined with feeding troughs for delivering and mixing predetermined amounts of different materials, *e.g.*, dry food for live-stock. B. M. VENABLES.

Process and apparatus for the production of cold. PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (E.P. 238,223, 28.7.25. Conv., 8.8.24).—A cooler, an evaporator, and a combined condenser and absorber are connected for closed-cycle operation. The gaseous refrigerant is separated with absorption of heat from the solution in the cooler, the separation being assisted by circulating an auxiliary medium between the evaporator and the cooler. The separated gas is delivered to the absorber, and, without undergoing condensation, is reabsorbed in solvent condensed from vapour received from the evaporator, and this solution overflows into the cooler. The circulations are maintained under the slight pressure differences resulting within the system. With ammonia as refrigerant, the solvent may be water and the auxiliary medium a concentrated alkaline solution, preferably a mixture of sodium and potassium hydroxides. With carbon dioxide as refrigerant, the solvent is sulphur dioxide and the auxiliary medium is carbon disulphide. Brine may also be used as auxiliary medium. Circulation of the refrigerant through the boiler may be prevented by expelling the refrigerant from the cooler through a gas separator inserted in the circulatory system of the auxiliary medium, and preferably heated by liquid from the absorber. An additional refrigerating effect may be obtained by evaporation of the solvent in the cooler, the vapour being withdrawn and absorbed by the auxiliary medium in a vessel separate from the main absorber. H. HOLMES.

Concentration of liquids by vaporisation at very low temperatures. P. C. Lemale (F.P. 609,295, 16.4.25).—Steam at a temperature much below 100° is employed and, by means of an ejector–condenser, two stages of vacuum are produced, the first being established in a heating device traversing the apparatus, the other in the evaporator. J. S. G. Thomas.

Emulsifying agents. I. G. Farbenind. A.-G. (F.P. 608,302, 22.12.25. Conv., 18.9.25).—Permanent emulsions of fats, oils, waxes, hydrocarbons, etc. are obtained by the use of glue, gum arabic, etc. in conjunction with usual emulsifying agents, *e.g.*, soaps, sulphonic acids, etc. S. S. Woolf.

Centrifugal separating machines. K. J. Svensson and K. A. P. Norling (E.P. 264,777, 7.12.26. Conv., 10.9.26).—In a centrifugal separator for two liquids which is provided with conical separating plates with holes in them for the upward flow of the mixture, it is desirable that the incoming mixture should be admitted approximately on the line of separation of the two liquids. To effect this the bottom distributing plate is either made so as to be easily changed and provided with several holes at different radii, or the distributing plate is arranged to turn in relation to the plate next above it, one of the plates being provided with radial series of holes or slots, and the other with non-radial inclined slots, so that on turning one in relation to the other the common openings in them will vary in distance from the axis. B. M. Venables.

Fluid separators. E. C. R. Marks. From Tracy Engineering Co. (E.P. 265,250, 6.8.25).—The fluid is settled in a chamber or conduit having an elongated inlet, which is wide at one end but converges towards the outlet end. The chamber may also be provided with easily detachable collecting baffles. B. M. Venables.

Apparatus for expressing liquid constituents from materials of various kinds. F. Krupp Grusonwerk A.-G. (E.P. 252,005, 6.5.26. Conv., 7.5.25. Addn. to E.P. 248,372; B., 1927, 176).—The inner and outer strainer-walls of the annular press chamber are subjected to a relative rotary tilting movement by an eccentric associated with the means for driving the rotary finger which forces in the material. The finger moves outwards against an adjustable spring under the pressure in the chamber, and this movement is communicated to rotary discharging knives. H. Holmes.

Purifying, cooling, mixing, absorbing gas, air, vapours, etc. Allgem. Vergasungs-Ges.m.b.H. (F.P. 609,180, 20.11.25. Conv., 19.12.24).—The washing or absorbing liquid flows in counter-current to the gas stream through chambers containing rotating discs supported by plates etc. J. S. G. Thomas.

Method and apparatus for adsorbing a gas or vapour from mixtures thereof. Silica Gel Corp., Assees. of E. C. Holden (E.P. 257,879, 22.10.25. Conv., 4.9.25).—The adsorbent is placed on a number of trays in an enclosing casing; the spaces between the trays opening alternately on one side to the inlet and on the other side to the outlet. The trays may be inclined to each other so that the spaces between are wedge shaped, the large ends being the inlets or outlets. The saturated adsorbent is recovered later by passing hot gases of combustion through it. In the case of, *e.g.*, blast-furnace gas which has been freed from water vapour by the adsorbent, a portion of the same gas may be burnt and, if necessary, diluted with cleaned air and used to dehydrate the absorbent. B. M. Venables.

Plants for carrying out gaseous catalytic reactions at high temperature and pressure. G. L. E. Patart (E.P. 252,362, 12.5.26. Conv., 25.5.25).—The catalyst is contained in units each comprising a horizontal pipe or vessel which is closely wound with a smaller pipe conveying the reacting fluid to the catalyst. The whole is heated by a row of external gas flames, so that the catalyst is heated indirectly and uniformly both by means of the heated fluid and by conduction through the wall of the vessel. Additional means may be provided for preheating the fluid by the products of combustion. The catalyst is charged in cartridge form, and is preferably clamped tightly between the lids of the vessel, so that it completely fills the vessel. B. M. Venables.

Determining the dust content of gases. Siemens-Schuckert-Werke G.m.b.H. (F.P. 609,988, 26.1.26. Conv., 24.8.25).—The gas is delivered between two insulated plates and the alteration of the capacity of the plates determined. J. S. G. Thomas.

Apparatus for atomising liquids in rotating gas-purifiers, absorption plant, gas mixers, or coolers, etc. E. Theisen (G.P. 434,605, 25.3.24).—One or more worm-wheel atomisers are used in conjunction with one or more hollow cylindrical or conical perforated atomisers. A longitudinal-screw vane operates outside the perforated cylindrical or conical jacket of the atomisers. J. S. G. Thomas.

Liquefaction and fractionation of gas mixtures, more especially air. R. K. E. Mewes (F.P. 610,024, 2.5.25. Conv., 16.6.24, 4.7.24, 15.8.24, 18.11.24, and 16.2.25).—The gas mixture, more especially air, is introduced into the fractionating plant at a pressure of 0—15 atm., while one fraction, *e.g.*, preferably nitrogen, in the case of air, circulates under high pressure in order to produce the necessary compensating coldness. In certain cases, air compressed under high pressure is employed. J. S. G. Thomas.

Separation of organic gases from admixture with gases not readily absorbed. I. G. Farbenind. A.-G., Assees. of A. Engelhardt (G.P. 435,013, 11.12.20. Addn. to G.P. 310,092; B., 1921, 204 a).—Charcoal from which sulphur has been removed is used in the earlier process. L. A. Coles.

Storing and transporting gases. Union Générale Co-opérative (E.P. 252,385, 20.5.26. Conv., 20.5.25).—The gas is stored in a vessel which is contained in a second vessel having heat-insulating walls and containing in the space between it and the first vessel a second gas in liquid form boiling at a lower temperature than the gas in the inner container, which is thus constantly kept cool. R. A. A. Taylor.

Drying apparatus. M. M. Minter (E.P. 265,449, 18.6.26).—See U.S.P. 1,594,316; B., 1926, 808.

Methods and apparatus for heating materials.

Leeds & Northrup Co., Assees. of J. W. Harsch (E.P. 247,912, 9.9.25. Conv., 20.2.25).—See U.S.P. 1,578,027; B., 1926, 446.

Apparatus for continuous filtering. W. D. Mount (E.P. 265,679, 12.8.25).—See U.S.P. 1,558,038; B., 1925, 976.

[Fluid-cooled wall for] furnaces. T. E. Robertson. From Power Specialty Co. (E.P. 265,422, 10.4.26).

Water-proof and dust-proof sack or like container for conveying granular, dusty, and brittle materials. Felten & Guilleaume Carlswerk A.-G. (E.P. 261,427, 15.11.26. Conv., 14.11.25).

[Nozzles etc. for the] atomisation and desiccation of liquids and solutions. Nestlé & Anglo-Swiss Condensed Milk Co. (E.P. 265,840, 15.6.26. Conv., 23.2.26).

Cooling plates. F. B. Dehn. From A. Jacobi A.-G. (E.P. 265,845, 22.6.26).

Concentration of materials (E.P. 265,252).—See VII.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Hydrogenation and liquefaction of coal. IV. Hydrogenation of cannel coal. D. G. Skinner and J. I. Graham (Fuel, 1927, **6**, 74—81).—Using the method previously described (B., 1925, 947), the results of hydrogenation of a Fifeshire boghead coal from the Lower Dysart seam (ash 25·82%, moisture 1·07%, volatile matter 55·58%) and a South Staffordshire Thick cannel coal (ash 3·35%, moisture 5·20%, volatile matter 46·07%) were compared with those from the durain of the foregoing thick coal (ash 3·50%, moisture 6·06%, volatile matter 31·18%). In a preliminary carbonisation assay less water was evolved from the boghead coal than from the other samples. At 900° the cannel yielded a harder coke than the durain, whereas the boghead coal had no coking properties. Extraction with pyridine at 72° *in vacuo* yielded 2·95% for the boghead coal and 16·7% for the cannel. The oil yield per ton at 600° using the assay apparatus was 99 gals. for the boghead coal, 54 gals. for the cannel, and 24 gals. for the durain. Hydrogenation at 430° and 120 atm. yielded 94, 79, 80 gals. respectively. The total production of oil from boghead coal is not increased by hydrogenation as in the other cases, but the proportion of constituents volatile below 300° is increased and the iodine value reduced. Using nitrogen instead of hydrogen in "blank" experiments, lower yields of oil were obtained than in the assay. Owing to similarity in composition between Scottish oil shales and boghead coals, hydrogenation will not materially increase the oil yield from the former substance. A. C. Monkhouse.

Study of the swelling power of coal at different rates of carbonisation. L. Slater (Fuel, 1927, **6**, 82—84).—Cokes produced in the normal volatile-matter tests are unsatisfactory as a basis of comparison with large-scale practice. Ten coals were examined in an assay apparatus carbonising 20 g. of coal; the silica retort was introduced into the furnace at 300°, the temperature raised to 600° in 1 hr. and maintained at 600° for 1 hr. A division of the coals according to degree of swelling and physical appearance of the coke was made; in certain cases a better differentiation was obtained by increasing the period of heating to 4½ hrs. The test is satisfactory for a comparison of coals from one geological area, but is insufficient for the evaluation of coals from different fields. It is also suitable as a guide in the choice of coals for use in gas-producers. A. C. Monkhouse.

Selection of coals for the manufacture of coke. H. J. Rose (Fuel, 1927, **6**, 84—88).—The majority of the coals used in by-product ovens in America are from the States of Pennsylvania, W. Virginia, Alabama, and Kentucky. Cokes for blast furnaces should not contain more than 12% of ash, 1·3% S, and 0·01% P; they should be of medium density, with fairly large cells and thin or porous cell walls, uniform in size; the largest possible percentage should lie between 2 in. and 4 in. sizes of coke. Foundry coke should be of large size, good strength, and have a minimum reactivity with carbon dioxide; it should contain not more than 2% of volatile matter, 12% of ash, 1% S, and not under 86% fixed C. The best results are obtained by blending coals of low and high volatile content, and by using low temperatures of carbonisation and long coking times. Domestic coke should be a screened, dense coke with an ash content of 8—9% and a fusing temperature of above 1200°. Water-gas coke should be from 2 in. to 4 in. size, of low sulphur and ash content, and have an ash fusing at 1310—1370°. A. C. Monkhouse.

Carbonisation of particles of coal. Cenospheres. III. H. E. Newall and F. S. Sinnatt (Fuel, 1927, **6**, 118—120; cf. B., 1926, 905).—Determinations have been made of the changes in volatile matter content and agglutinating value of particles of coal (60—90 mesh) which were allowed to fall repeatedly through a heated tube containing nitrogen. The times of heating required to destroy completely the caking powers of three coals of initial agglutinating values 10·0, 8·5, and 4·0, were 25·2, 21·6, and 12·0 sec. respectively at 400° (taking 1·2 sec. as the approximate time of fall through the heated zone 18 in. in depth), the corresponding losses of volatile matter being 6·6, 3·9, and 6·1%. At 600° only about 3 sec. was required to destroy the caking powers of the same coals, but the volatile matter evolved per unit loss of agglutinating value was higher. Larger coal particles (10—30 mesh) suffered very little change in properties even after 13 treatments at 600°. The behaviour of the alpha, beta, and gamma fractions of one of the coals on passage through the heated tube was examined; the cenosphere form could be observed in the product from the gamma fraction, but not from the others. A. B. Manning.

"Pinking" in internal-combustion engines. G. B. Maxwell (Fuel, 1927, **6**, 121—130).—The various theories that have been proposed to account for "pinking" in internal-combustion engines, and for the action of "anti-knock" substances, are critically reviewed. Callendar's nuclear hypothesis (cf. preceding abstract) appears to fit the facts better than any other, but requires further research, in particular on the question of peroxide formation and the action of "knock inducers," to substantiate it fully. A. B. Manning.

Autoxidation and antioxygenic action. XXI. Application to the mode of action of antidetonants. C. MOUREU, C. DUFRAISSE, and R. CHAUX (Compt. rend., 1927, **184,** 413—417; cf. A., 1926, 581).—Autoxidation, or the union between an oxygen molecule and that of an autoxidisable substance to form a primary peroxide with absorption of energy, has been investigated at 160° for hydrocarbons and animal and vegetable oils. The paraffinic hydrocarbons show an autocatalytic autoxidation preceded by a period of induction which is suppressed by the addition of certain substances. The autoxidations of tetrahydronaphthalene and of most animal and vegetable oils are higher than those of paraffin, but decahydronaphthalene, naphthalene, and copra oil are less oxidisable. The formation of peroxides has been recognised in a number of petroleum fractions. The effects of various amounts of a number of organic compounds on the speed of absorption of oxygen have been noted in each of the above cases. Antidetonants act as antioxygens in the liquid rather than in the gaseous phase of the explosion mixture, and the formation of peroxides precedes all autoxidation. Antidetonants exert their effect by the prevention of the formation of peroxides, but an increase in the concentration of oxygen increases the intensity of peroxidation.

J. GRANT.

Gaseous explosions. Effect of fuel constitution on rate of rise of pressure. G. G. BROWN and G. B. WATKINS (Ind. Eng. Chem., 1927, **19,** 280—285).—The rate of rise of pressure on the explosion of various compounds used as liquid fuels with the theoretical quantity of air was determined. To the bomb used was connected a "Midgley indicator," in which the compression of a spring rotated the axis of a concave mirror. This mirror threw a beam of light on to photographic paper on a revolving drum, the source of light and the explosion spark being actuated by closing the same electric circuit. The gaseous products of combustion were analysed. The pressure element was calibrated by means of a dead-weight tester. The average value of dP/dt was found to increase with mol. wt. in the paraffin series, to decrease with addition of methyl groups to the benzene ring, and to be about the same for higher alcohols as for the corresponding paraffin hydrocarbons. Typical figures are: *n*-heptane 37,400 lb./sq. in./sec., benzene 43,400 lb./sq. in./sec., and ethyl ether 48,400 lb./sq. in./sec. In view of general experience, it is evident that this quantity dP/dt is not in itself the measure of the tendency of a fuel to "knock" in an internal combustion engine.

C. IRWIN.

Ignition of gases. VI. Ignition by flames. Mixtures of the paraffins with air. N. S. WALLS and R. V. WHEELER (J.C.S., 1927, 291—297).—The relative ease of ignition of mixtures of methane, ethane, propane, and pentane, severally, with air was determined by measuring the time required to effect ignition when the mixtures were exposed to a fully-aerated flame. It was found with each inflammable gas that the mixture with air that required the shortest time of exposure was that in which the initial speed of propagation of flame is fastest. It is concluded that the ease of ignition by a momentary source of heat is dependent essentially on the normal speed of propagation of flame in the mixture. The apparatus devised for the measurements is described.

W. THOMAS.

Dehydration of manufactured gas. F. W. SPERR, JUN. (Chem. Met. Eng., 1927, **34,** 97—100).—Gas is dehydrated by multi-stage compression with intermediate cooling between the stages. A compression of 50—80 lb./sq. in., and expansion into the mains at 5 lb./sq. in. to yield a gas with a dew point of —1·1° is satisfactory. The method is expensive on account of the high power cost, but naphthalene is also removed. Refrigeration to remove moisture can be effected by a two-stage system, first cooling to 3·3—4·4° by direct-contact water, and then to —1·1° with brine-cooled coils. Refrigerated brine for direct contact is unsatisfactory owing to the entrainment of minute droplets, and also because means must be provided to concentrate the brine. The chief item of expense is the power needed, and this, by the compression system, is greatly reduced by supplemental refrigeration, using water cooled to 1·1—1·8°. Curves are given showing the power requirements for cooling from various initial temperatures. Hygroscopic substances such as calcium chloride or sulphuric acid may be used, and with these the removal of light oils is reduced to a minimum, but the temperature of the system must be kept down and the entrainment of the drying agent in the gas prevented. Solid calcium chloride has disadvantages, but a saturated solution at 15·5° is theoretically capable of reducing gas to a dew point of nearly —1·1°, and with artificial cooling it is possible to dehydrate satisfactorily. Concentrated sulphuric acid is most effective, or acid of *d* 1·70 is suitable if some cooling is employed. Silica gel may be applied to the dehydration of gas by adsorption, and although benzol is also adsorbed it is gradually replaced by the water. The author states that the methods given could probably be applied at trifling cost, and water-sealed holders could be used if a layer of oil were added.

W. G. CAREY.

Cyanogen in gas and the economics of the wet cyanogen purification process with regard to the present-day higher demands of purity of gas for household and industrial use. R. MEZGER (Gas- u. Wasserfach, 1927, **70,** 165—171).—Methods of diminishing corrosion by a more complete removal of the active impurities in coal-gas present fewer practical difficulties than the proposed American method of drying the gas (cf. preceding abstract). A full analysis is made of the economics of cyanogen removal by Bueb's process, based on the practice of the Stuttgart gas-works. The gas contains 0·5% H_2S and 115 g./m.^{3} of hydrogen cyanide, 21% of the latter remaining in the gas after iron oxide purification and ammonia scrubbing. The cost of oxide purification, without the previous removal of cyanogen, is estimated; complete cyanogen removal results in a 21% lowering of the cost of the subsequent oxide purification, due to increased efficiency of the latter, no oxide being utilised for the formation of Prussian blue, and to the removal of part of the sulphur in the cyanogen washer. In addition to this the cyanogen recovery itself results in a net gain. These gains, however, are small compared with the indirect advantages

due to the diminution of corrosion in the pipes, holders, etc. A. B. MANNING.

Action of gaseous hydrogen iodide on petroleum derivatives containing sulphur, and on some organic compounds, especially thiophen. F. J. NELLENSTEYN (Chem. Weekblad, 1927, **24**, 102—105. Cf. B., 1924, 285; 1925, 33).—Aliphatic sulphur compounds do not react with dry hydrogen iodide. Aromatic compounds containing sulphur evolve hydrogen sulphide more or less readily. Thiophen reacts at once, even in cold dilute solution, with separation of carbon, iodine, and hydrogen sulphide. An aqueous solution of hydrogen iodide only reacts with thiophen if the mixture is heated in a sealed tube at 140°. The reaction occurs in two stages: in the first, sulphur is removed from the ring; in the second, the liberated iodine attacks the hydrocarbon residue. The separated carbon contains much adsorbed iodine and sulphur. Furan and methylfuran act in the same way. The treatment may be used to reduce the sulphur content of petroleum distillates; in one experiment a 20% reduction was observed. S. I. LEVY.

Synthesis of petroleum. F. FISCHER (Fuel, 1927, **6**, 89—94).—Recent work on the hydrogenation of coal and carbon monoxide is reviewed with special reference to the manufacture of synthetic alcohols (synthol) and the production of petroleum hydrocarbons by hydrogenation of carbon monoxide at ordinary pressure. A. C. MONKHOUSE.

Apparatus for determining the softening point of paraffin and other waxes, pitches, etc. H. HERBST (Chem.-Ztg., 1927, **51**, 140—141).—A brass rod resting on the surface of the wax etc. under test is attached to a balance weight by a thread passing over a fixed pulley, an indicator needle on the spindle of the latter recording on a dial any movements of the rod. The wax, contained in a securely clamped tube, is heated in a double heating bath, and observations of the progressive softening can be made as the rod slowly sinks. S. S. WOOLF.

PATENTS.

Coal washing and ore concentration. T. M. CHANCE (U.S.P. 1,605,172, 2.11.26. Appl., 26.7.24).—In the application of the method of ore flotation, coal-washing, etc., described in U.S.P. 1,462,881 (B., 1923, 962 A), a better separation of the light and heavy material is obtained by maintaining an accumulation of the heavy material in the bottom of the washing-vessel, in the path of the entering washing water, which it serves to disperse over the whole cross-section of the washing vessel, effectively preventing all central streaming of the liquids. T. S. WHEELER.

Apparatus and method for the distillation or heat treatment of carbonaceous and other materials. F. D. MARSHALL (E.P. 261,919 and 261,975, 5.6.25).—(A) A vertical retort, the horizontal section of which is in the form of two intersecting circles, is provided with two screws which revolve about vertical axes passing respectively through the centres of the two lobes of the retort. The screws are spaced apart, so that, although their threads revolve within one another, they are not in contact. This spacing of the screws allows the free passage of the gaseous distillation products, or of a gaseous distillation medium, through the retort. The co-operating screws, which are rotated preferably in the same direction, force the material undergoing distillation through the retort in the form of segmental rings disposed between the threads of the screws. The thickness of these rings to ensure rapid and complete carbonisation is $4\frac{1}{2}$ in. (B) The retorts are heated by the combustion of the hot "blow" gases from one or more water-gas generators. The "blow" gases are freed from dust in a cyclone extractor. The water-gas may be superheated by circulation round the retorts and then passed into the retorts so as internally to carbonise the material. S. PEXTON.

Distillation or heat treatment of carbonaceous and other materials and manufacture of briquettes. F. D. MARSHALL (E.P. 261,927, 5.6.25).—A retort of the form described in the preceding abstract is superimposed upon a heating chamber which contains heating members arranged radially or transversely. The screws operating within the retort force the partially carbonised product through the spaces intervening the heating members. The shape of the briquetted product is in section that of the spaces intervening the heating members in the lower chamber. S. PEXTON.

Production of carbon. A. LEDERER (E.P. 251,244, 15.3.26. Conv., 24.4.25).—Carbon is formed by the thermal dissociation of carbon disulphide. The separated sulphur may, however, attack the substance of the tubular reaction vessel, and form compounds contaminating the carbon. Immediately upon passing through the decomposition zone of the furnace the vapours are therefore subjected to a sudden temperature drop. The decomposition zone may be formed by an electrically-heated device suspended axially in the tube, and made of or coated with a substance inert to the vapours from the decomposition. The whole tube may then be cooled from the outside. A pressure drop may also be induced in the reaction vessel. R. A. A. TAYLOR.

Treatment of charcoal. RÜTGERSWERKE A.-G., and L. KAHL (G.P. 434,269, 14.9.23).—Charcoal having low porosity and high density and electrical conductivity is prepared by impregnating ordinary charcoal with tar, tar oil, or pitch which has previously been subjected to the action of ozone, oxygen, or gases containing oxygen, under the usual reduced or increased pressure, and, if necessary, in the presence of catalysts, and repeatedly calcining the product. Alternatively, the treatment with ozone or oxygen may be effected at a raised temperature after impregnation. L. A. COLES.

Manufacture of coal gas. SOUTH METROPOLITAN GAS CO., and E. V. EVANS (E.P. 265,349, 18.12.25).—On steaming a charge in a high-temperature retort the calorific value of the total gas made is reduced by water-gas entering into its composition. The calorific value is therefore raised to the required standard by addition to the high-temperature gas of low-temperature gas made in a setting heated by waste heat from the high-temperature setting. R. A. A. TAYLOR.

Water-gas. COMBUSTION RATIONELLE (F.P. 609,340, 23.4.25).—In the decomposition of steam by a suitable powdered fuel at high temperatures the fuel is raised

to the necessary temperature by means of air rich in oxygen or by oxygen itself. A. B. Manning.

Desulphurising natural or artificial gas mixtures. Soc. Internat. des Procédés Prudhomme (S.I.P.P.) (F.P. 609,678, 25.4.25).—The gas streams over metallic suboxides or oxides, or over more or less oxidised metals, *e.g.*, nickel. J. S. G. Thomas.

Dust separator for carbonisation gases. Thyssen & Co., A.-G., Assees. of R. Truschka (G.P. 434,352, 1.6.24).—The gas is passed through a filtering chamber containing coke or similar material, which is heated to prevent condensation of tar vapours thereon. To avoid danger of explosion, the heating is carried out electrically or by heated metal or high-boiling oil baths in the walls of the filtering chamber. A. B. Manning.

Process and apparatus for the fractional distillation at a low temperature of solid fuels. A. Meiro (E.P. 265,435, 12.5.26).—The fuel to be carbonised is carried in finely-divided form through a system of retorts by means of an oil having a b.p. higher than the temperature of evolution of the volatile products from the fuel. The mixture of oil and fuel is passed through the retorts by means of a conveyor, and the retorts may be arranged in series and heated progressively. The agitation of the charge may be supplemented by blowing a gas through the liquid layer.
R. A. A. Taylor.

Obtaining low-boiling hydrocarbons and other products from distillation products derived from bituminous materials. Braunkohlen-Produkte A.-G., K. Bube, and E. Erlenbach (E.P. 265,375, 3.2.26).—The distillation products, being oxygenated and consequently poor in hydrogen, are mixed with a heavy petroleum distillate rich in hydrogen. The mixture is then cracked, the vapours interact, and the hydrogen in the products is evenly distributed. Catalysts (*e.g.*, of aluminium or magnesium) may be used.
R. A. A. Taylor.

Production of viscous oils from cracked distillates. Braunkohlen-Produkte A.-G. (F.P. 608,425, 28.12.25).—The cracked distillates are treated with anhydrous aluminium chloride at temperatures not higher than 100°. A. B. Manning.

Decomposition of heavy oils. H. Hernu (F.P. 608,332, 23.12.25).—The solid, liquid, or gaseous hydrocarbons in the reduction zone of a producer in operation are passed into contact with incandescent carbon, in the presence of nascent carbon monoxide and hydrogen. A mixture of permanent gases which can be used for driving motors is thereby produced. A. B. Manning.

Continuous cracking of bituminous materials. Braunkohlen-Produkte A.-G. (F.P. 608,422, 28.12.25).—The starting material, *e.g.*, lignite, is mixed with liquid hydrocarbons and the resulting paste pumped continuously through an autoclave heated to above 300°.
A. B. Manning.

Distilling and refluxing unit [for oil]. H. H. Armstrong (U.S.P. 1,616,840, 8.2.27. Appl., 26.12.23).—The still is supported in a furnace above a pre-heating coil, and the oil, delivered through the coil, enters the upper front portion of the still by way of a spray-producing device. The rear upper portion of the still communicates directly with a dephlegmator from which the condensates are returned to the still in a fine spray. Means are provided for withdrawing vapours from the top of the dephlegmator, and for withdrawing the residual oil from the lower rear portion of the still.
H. Holmes.

Cracking hydrocarbon oils. G. Egloff and H. P. Benner, Assrs. to Universal Oil Products Co. (U.S.P. 1,617,619, 15.2.27. Appl., 21.8.20. Renewed 9.6.26).—The oil is heated to the desired temperature in a pipe still, it passes thence to a vertical separating chamber, where it passes downwards, then upwards, liquid oil and precipitated carbon being drawn off from the bottom of the chamber through a rotary valve, and vapours are taken off at the top and condensed.
W. N. Hoyte.

Purification of benzines etc. produced by cracking. I. G. Farbenind. A.-G., Assees. of C. Wegner (G.P. 433,855, 17.12.24).—Water-white, odourless, and non-gumming benzines are obtained by treating the oils with moist ferric chloride. A. B. Manning.

Purification of hydrocarbons. J. Demant (Addn. No. 30,617, 12.6.25, to F.P. 530,490).—The procedure of the main patent is applied to heavy tar oils, shale oils, and mineral oils boiling above 200°, giving highly purified products which do not polymerise under the influence of heat or other agents. A. B. Manning.

Motor spirit. E. P. Dumanois (F.P. 609,336, 23.4.25).—A motor spirit consists of a mixture of light petroleum, heavy hydrocarbons, benzine, and an "anti-knocking" agent, *e.g.*, lead tetraethyl, in such proportions that the light petroleum and the heavy hydrocarbons form at least 20% of the mixture.
A. B. Manning.

[Treatment of fuel] alcohol and its homologues and mixtures containing the same. Benzol-Verband G.m.b.H. (E.P. 257,881, 14.12.25. Conv., 4.9.25).—The corrosive action of fuel alcohols and benzol–alcohol mixtures on metals is counteracted by the addition of less than 1% (preferably 0·2—0·3%) of a salt of benzoic acid, in particular the sodium salt.
A. Davidson.

Recovery of oil from emulsions. H. N. Dons, Assr. to N. A. Hallauer (U.S.P. 1,617,201, 8.2.27. Appl., 20.6.22).—Cellulose impregnated with caustic alkali is mixed with the emulsion, whereby separation of the oil from the water and foreign matter is effected.
W. N. Hoyte.

Use of a sludge-laden liquid in petroleum or gas wells. National Pigments & Chemical Co. (F.P. 610,800, 10.2.26).—A sludge is prepared from barium salts, ferric oxide, or litharge, or these substances are added to a light clay sludge, and the product used in boring or controlling the oil or gas sources.
A. B. Manning.

Production of coke and semi-coke. E. B. G. Bascou, Assr. to Soc. Anon. des Petroles, Houilles, et Dérivés (U.S.P. 1,618,563, 22.2.27. Appl., 4.6.24. Conv., 28.11.23).—See E.P. 225,505; B., 1925, 345.

Furnace and apparatus for utilising shale or other bituminous materials. S. V. Bergh (U.S.P. 1,618,566, 22.2.27. Appl., 25.9.23. Conv., 18.9.22).—See E.P. 204,067; B., 1925, 90.

Manufacture of vegetable carbon. L. H. BONNARD, Assr. to A. H. BONNARD (U.S.P. 1,619,649, 1.3.27. Appl., 8.10.25. Conv., 4.8.25).—See E.P. 260,666 ; B., 1927, 34.

Apparatus for and process of manufacture of combustible gas. I. C. COPLEY, Assee. of W. J. MURDOCK, E. E. LUNGREN, and O. B. EVANS (E.P. 240, 440, 10.9.25. Conv., 23.9.24).—See U.S.P. 1,602,242 ; B., 1927, 35.

Distillation and carbonisation retort and process of operating the same. F. DUPLAN (U.S.P. 1,618,865, 22.2.27. Appl., 15.10.23. Conv., 20.10.22).—See E.P. 225,337 ; B., 1925, 90.

Process of producing lubricating oils. G. A. KRAMER, Assr. to SIMPLEX REFINING Co. (U.S.P. 1,619,348, 1.3.27. Appl., 9.5.25).—See E.P. 251,928 ; B., 1926, 908.

Fuel burners. INTERNAT. COMBUSTION LTD., and F. H. ROSENCRANTS (E.P. 265,410, 19.3.26).

Tanning agents (G.P. 433,162).—See XV.

III.—TAR AND TAR PRODUCTS.

Cause of the red colour of aqueous extracts and emulsions of low-temperature tars. S. P. BURKE and S. CAPLAN (Ind. Eng. Chem., 1927, **19**, 34—38).—The material in low-temperature tars which is responsible for the red colour of their aqueous extracts and of disinfectant emulsions prepared from their distillates has been isolated by extraction of the tar distillate with a saturated solution of borax, and subsequent distillation *in vacuo* of the black viscous oil obtained. The colouring material, which constitutes about 0·8% of the tar, is a mixture of *o*-dihydric phenols, including homologues of pyrocatechol but not pyrocatechol itself. The predominating compound is a homologue of pyrocatechol which has not previously been described, viz., either 3 : 4- or 3 : 6-*dimethylpyrocatechol*, b.p. 253° (corr.), m.p. below —20°, d_4^{25} 1·1426, n_D^{20} 1·5609 ; it yields an *acetyl* derivative, b.p. 281°, and a *nitro*-derivative, m.p. 108°. (Cf. Mcleod and others, B., 1927, 37.)

A. B. MANNING.

Softening point of pitch etc. HERBST.—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Lake dyestuffs and their application. A. W. C. HARRISON (J. Oil Colour Chem. Assoc., 1927, **10**, 28—37). —An account of some of the difficulties met with in the conversion of dyestuffs into lake pigments. Dyestuffs are classified as water-soluble dyestuffs (basic and acidic), slightly soluble acidic or insoluble dyestuffs requiring a metallic precipitant, and insoluble dyestuffs not requiring any metallic precipitant, and the procedure for each type is discussed. S. S. WOOLF.

PATENTS.

Manufacturing [perylene] dyes. W. PENECKE, Assr. to F. BENSA (U.S.P. 1,615,646, 25.1.27. Appl., 7.4.25. Conv., 10.4.24).—Perylene is heated with 2—3 pts. of phosphorus tri- or penta-sulphide at 230° to yield a *product* which dyes wool in acetic acid suspension, and cotton in alkaline solution, red shades, and which, on treatment with alkaline hydrogen peroxide, gives a *substance* which dyes wool orange shades, and on heating with sodium sulphide and sulphur at 300° yields a *product* which gives on cotton moss-green shades which are fast to light. T. S. WHEELER.

Trisazo dyestuff. Tetrakisazo dyes. J. B. OESCH, Assr. to NEWPORT Co. (U.S.P. 1,615,550—1, 25.1.27. Appl. [A, B], 6.2.26 ; cf. 1,605,973—4 ; B., 1927, 135).—(A) Diazotised acetamido-Cleve's acid (4-acetamido-1-naphthylamine-6- and -7-sulphonic acids) or (B) a diazotised aminoazo-dyestuff, *e.g.*, 2 : 3′-dimethylazobenzene-4 : 6′-disulphonic acid, is coupled with *m*-toluidine, the product is diazotised and coupled with *m*-toluidine, and this product is diazotised and coupled with 1-phenyl-3-methyl-5-pyrazolone, or its *p*-sulphonic acid, to give *substances* which dye unmordanted cotton orange to brown shades, very fast to light.

T. S. WHEELER.

Production of anthraquinone derivatives. H. A. E. DRESCHER, W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 264,916, 21.7.25 and 21.5.26).—The α- and β-aminoanthraquinone derivatives formed by cyclisation of 3-aminobenzoyl-*o*-benzoic acids containing also a substituent (X) in the 4-position are separable by fractional precipitation from sulphuric acid or fractional crystallisation from concentrated hydrochloric acid or from organic solvents. When X = Cl there are obtained 3-chloro-2-aminoanthraquinone, m.p. 310—311·5°, and 2-chloro-1-aminoanthraquinone, m.p. 196—197°. Bromination of the mixed chloroaminoanthraquinones gives a mixture of 2-*chloro*-4-*bromo*-1-*aminoanthraquinone*, m.p. 219°, and 3-*chloro*-1-*bromo*-2-*aminoanthraquinone*, separable in the same way ; chlorination yields 1 : 3-*dichloro*-2-*aminoanthraquinone*, m.p. 227·5—228°, separable from the corresponding 2 : 4-dichloro-1-aminoanthraquinone. When X = Br, 3-bromo-2-aminoanthraquinone, m.p. 305—306·5°, is separated from the 2 : 1-isomeride, or the mixture may be brominated to give 2 : 4-dibromo-1-aminoanthraquinone, m.p. 203—215°, and 1 : 3-dibromo-2-aminoanthraquinone. When X = Me there is obtained 2-*amino*-3-*methylanthraquinone*, m.p. 259—260°, which on bromination gives 1-*bromo*-2-*amino*-3-*methylanthraquinone*, m.p. 192—193° ; 1-amino-2-methylanthraquinone may be obtained from the acid filtrates of the 2 : 3-isomeride. C. HOLLINS.

V.—FIBRES ; TEXTILES ; CELLULOSE ; PAPER.

Structure and strength of aerated [hollow] artificial silks. A. HERZOG (Kunstseide, 1926, 397—401).—Hollow viscose artificial silk fibres (*e.g.*, Celta, Luftseide, Soie Nouvelle, etc.) produced by squirting viscose solutions which are aerated or contain substances capable of liberating carbon dioxide or other suitable gas during coagulation in an acid bath are less dense than normal solid viscose fibres, and owing to peculiar reflexion of light from both cuticle and lumen they have greater covering power when present in textile materials. During processes of production the air within the tubular fibres is displaced so that they collapse to a ribbon-like form somewhat resembling that of cotton. Those English, French, and German varieties of hollow fibres examined were of 4—5 denier,

and had average breaking strains (in dry state) of 1·52, 1·57, and 1·01 g./denier, respectively; when moistened they suffered a loss in tensile strength of 68·4%, 66·9%, and 69·9% respectively (corresponding to a loss of 68% for similar solid fibres). The extensions at breaking point of hollow fibres were 15—19% (dry) and 15—20% (wet), these being greater than the corresponding extensions of 13·8% (dry) and 15% (wet) for solid fibres. A. J. Hall.

Effect of temperature upon the freeness of sulphite pulp. D. S. Davis (Ind. Eng. Chem., 1927, **19**, 162—165).—The increased freeness of sulphite stock at raised temperatures is almost entirely due to the decreased viscosity of water and possibly, to a very slight extent, to dehydration of the fibre. Stock that has been heated shows on cooling its original freeness without any appreciable time lag. The rate of change of freeness with rise in temperature is higher for slow stock than for free stock, but in each case falls off as the temperature increases. From the graphs connecting freeness and temperature it is possible to calculate approximately the quantity of steam necessary to adjust the freeness of stock to the required value. Freeness–consistency charts intended for use at 20° are found to be applicable at higher temperatures for consistencies between 0·35 and 0·45%. D. J. Norman.

Freeness of groundwood pulp. D. S. Davis (Ind. Eng. Chem., 1927, **19**, 84—86).—Data are presented for groundwood pulp similar to those given previously for sulphite pulp (cf. preceding abstract and B., 1926, 660). Freeness–consistency curves for groundwood closely resemble those for sulphite stock. A freeness–consistency chart is given to obviate the necessity for working at exactly 0·4% consistency, and, as with sulphite pulp, this chart is reasonably accurate over a fairly wide temperature range. Between 10° and 50° the freeness of groundwood pulp is a linear function of the temperature, and it is shown that, when the viscosity of the suspending medium is constant, freeness increases with increasing temperature. Both sulphite stock and groundwood stock show slightly reduced freeness on keeping for several days. D. J. Norman.

Waste kauri wood as a source of paper pulp and resin (Bull. Imp. Inst., 1926, **24**, 654—664).—Various samples of waste kauri wood containing 50—60% of cellulose and 2—22% of resin were treated by the soda process before and after removing the resin by extraction with acetone or alcohol. The most satisfactory cooking conditions were 7 hrs. at 160° with 24% of caustic soda at a concentration of 4%. The untreated and extracted wood gave, respectively, 33—40% and 45—47% of a yellow-brown pulp which, though capable of conversion into paper of excellent strength, could not be satisfactorily bleached. The ultimate fibres averaged 4·5 mm. in length and 0·045 mm. in diameter. It is suggested that both waste and "fossil" kauri wood could be converted into paper of satisfactory quality, though, in the case of wood containing more than about 10% of resin, it would probably be necessary to extract and find a market for the resin. The properties of the resin, m.p. 70—73°, are described. D. J. Norman.

Production of currency paper. M. B. Shaw and G. W. Bicking (U.S. Bureau Stand. Tech. Paper No. 329, 1926, **21**, 89—108).—Details are given of experimental work carried out on a semi-commercial scale with a view of improving the wearing qualities of currency paper. Using new cotton and linen rags, the best furnish proved to be 75% of linen and 25% of cotton without any engine-sizing agents or fillers, these papers showing the highest folding test. The rags were digested in a rotary boiler at 30—40 lb./sq. in. for 5 hrs. with 6 or 12% (the latter for coloured linens) of lime, the ratio of liquor to rags being approximately 5 : 1. Bleaching was effected with calcium hypochlorite without the use of accelerators. The beating process, which determines to a large extent the strength of the paper, was carefully controlled by manipulating the beater roll to give a very gradual application of beater-roll pressure; 12 hrs.' beating at a consistency of 3·9% of pulp was found to be satisfactory. Waterleaf paper made under these conditions showed a folding test of 5000 in the machine direction and over 6000 in the cross direction. D. J. Norman.

Patents.

Increasing the strength and elasticity of vegetable fibres and products manufactured therefrom or for crinkling same. O. Dubac (E.P. 254,695, 15.6.26. Conv., 2.7.25).—The material is treated with a solution of sodium or potassium hydroxide (*d* 1·5 or over) at 60—100° for 1—5 min. or longer, the strength and elasticity of the fabric being thereby increased by as much as 150% and an appearance obtained resembling that of wool, silk, or linen according to the weave. The lustre is dull, and finishing with the usual additions is generally superfluous. No shrinking is shown on subsequent wetting or washing. B. P. Ridge.

Treatment of animal fibres. M. Bergmann, E. Immendörfer, and H. Loewe (U.S.P. 1,615,783, 25.1.27. Appl., 7.6.24. Conv., 23.2.23).—Animal fibres are protected during the removal of vegetable fibres by carbonisation with strongly acid reagents by conducting the operation in the presence of the active constituents of sulphite cellulose waste liquor. D. J. Norman.

Treatment of yarns and fabrics. British Celanese Ltd., and G. H. Ellis (E.P. 264,936, 30.10.25).—Degumming of natural silk in materials containing it mixed with cellulose acetate or other textile fibres is carried out without injury to the latter by impregnating the fabric with a mixture of sulpho-fatty acids (or their alkali or ammonium salts) and sodium silicate, carbonate, or borate, at 75°, allowing it to remain overnight, and subsequently scouring it with a soap solution containing sodium silicate, carbonate, or borate, at 75°.

B. P. Ridge.

Treatment of cellulosic materials and production of cellulose derivatives. H. Dreyfus (E.P. 263,938—9, 6.10.25).—(A) Cellulosic materials, preferably alkali treated as described in E.P. 249,173 (B., 1926, 436), are rendered more reactive for acetylation or other esterification by treatment with the vapours of lower fatty acids (*e.g.*, formic and/or acetic acids) either in the cold or with heating, such vapours being distributed throughout the cellulose, or finally removed from it, by

means of currents of air or indifferent gases. (B) Material pretreated as above and esterified in a quantity of acetic acid from 4 to 6 times the weight of cellulose requires only from under ½ to 3% of sulphuric acid on the weight of cellulose. The presence of 1—10% of substances such as ethylidene diacetate or lactic acid, which have an increased solvent power for the esterification products, prevents gelatinisation during acetylation.
B. P. RIDGE.

Manufacture of cellulose derivatives. H. DREYFUS (E.P. 264,937, 30.10.25. Cf. 207,562; B., 1924, 129, and 263,938, preceding abstract).—Cellulosic materials, pretreated according to the prior specifications, are esterified in the absence, or substantially in the absence, of benzol or other liquid diluents (so that the products are obtained in the fibrous or solid state instead of in solution) by passing the vapours of the esterifying agent alone, mixed with air or other indifferent gases, or with vapours of acetic acid or like solvents, over or through the material in the presence of condensing agents such as sulphuric acid. If necessary, the product is submitted to secondary treatment with the vapours in order to modify its solubility characteristics. B. P. RIDGE.

Apparatus for the manufacture of artificial silk. G. B. ELLIS. From SOC. POUR LA FABR. DE LA SOIE "RHODIASETA" (E.P. 264,708, 18.9.25. Cf. 233,384 and 248,696; B., 1925, 587; 1926, 945).—In spinning collodion through dies arranged within a tubular cell in which a gaseous medium causes evaporation of the solvent, part of this atmosphere is drawn off at a point intermediate between the ends of the cell, passed through a heating device, and, while still charged with the solvent vapours, re-introduced at a point near the dies. The auxiliary currents from several cells are mixed in one chamber, heated, and returned to the cells, solvent being recovered from the main current only.
B. P. RIDGE.

Manufacture of artificial silk etc. J. L. RUSHTON (E.P. 264,929, 29.10.25).—During the interruptions of winding by artificial silk spinning machines the delivery funnel is removed from the spinning box, inclined with its delivery end over a trough, and water passed through it thus carrying the filament to waste, thereby avoiding choking of the nozzle by cutting off the supply of solution and clogging of the "godet" rollers by the waste thread. The water supply is automatically controlled by movement of the funnel. B. P. RIDGE.

Combining cellulose and rubber. S. A. OGDEN (U.S.P. 1,617,495, 15.2.27. Appl., 1.8.23).—A mixture of rubber and cellulose material with sufficient sodium hydroxide to activate the cellulose is submitted to the action of steam, the excess sodium hydroxide removed, the product treated with acid to form hydrocellulose, and subsequently washed and dried. B. P. RIDGE.

Treatment of paper pulp stock. L. MELLERSH-JACKSON. From ROBINSON FIBER CORP. (E.P. 265,055, 1.6.26).—Sufficient sodium peroxide is added to the stock in the beater to make the mixture alkaline to litmus, and the beating is carried out at 49—54·5°. The resulting stock is clean, free-flowing, and does not deteriorate on keeping. Paper made from it is stronger and more uniform than that from untreated pulp, and remains white indefinitely. B. P. RIDGE.

Weighting of fibres. O. BERG and M. IMHOFF (E.P. 265,727, 2.12.25).—See U.S.P. 1,579,628; B., 1926, 485.

Mildew proofing of fibrous material. T. H. FAIRBROTHER and A. RENSHAW, Assrs. to BRITISH DYESTUFFS CORP., LTD. (U.S.P. 1,618,416, 22.2.27. Appl., 17.12.26. Conv., 24.7.25).—See E.P. 259,690; B., 1926, 1009.

Method and apparatus for the manufacture of artificial silk. F. W. SCHUBERT, Assr. to BRYSILKA, LTD. (U.S.P. 1,619,768—9, 1.3.27. Appl., 21.6.26. Conv., 2.7.25).—See E.P. 258,372 and 258,374; B., 1926, 1009.

Wool washing machines. F. B. PETRIE and PETRIE & MCNAUGHT, LTD. (E.P. 265,735, 14.12.25).

Apparatus for drying loose textile fibres. H. HAAS (E.P. 265,501, 16.10.26. Conv., 8.2.26).

Machine for wet treatment of textile fibres. C. FRÜH (E.P. 254,706, 25.6.26. Conv., 2.7.26).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Testing of wetting-out agents. W. KIND and J. AUERBACH (Textilber., 1926, **7**, 775—780).—A collection of results obtained in comparing proprietary wetting-out agents, by measurement of the drop-numbers and rates of sinking of standard pieces of fabric, in their aqueous solutions (cf. Auerbach; B., 1926, 705). The drop-numbers of 0·5% solutions (in distilled water) of Perpentol, Tetracarnite, Leonil S, Atmosol, Nekal A powder, Verapol, and Marseilles soap, increase in the order named, the drop-numbers being higher for solutions in hard water. The drop-numbers of solutions of 27 wetting-out agents increased slowly with rise of temperature at nearly the same rate; solutions of Marseilles soap, however, gave a much more rapid increase. The rates of sinking of fabric in 1% solutions of Oranit powder, Nekal A powder, Neomerpin N, and Leonil S in a wool-carbonising liquor (sulphuric acid of *d* 1·03) decreased in the order named, and this order of wetting-out powers is confirmed by the drop-numbers of the same solutions; the times taken by similar squares of fabric to sink in the carbonising liquor with and without the addition of 1% of Oranit powder were 3 sec. and 15 min. respectively. The wetting-out powers of 0·5% solutions of Nekal A, Neomerpin, Tetracarnite, and Transferin in a mercerising liquor (caustic soda of *d* 1·26) increased in the order named, the times of sinking of fabric in the mercerising liquor with and without the addition of 0·5% of Nekal A being 65 and 450 sec. respectively. The drop-number of a bleaching liquor containing a hypochlorite is increased by the addition of various wetting-out agents, but least by Perpentol. The penetration of bobbins of yarn when immersed in an aqueous solution of Diamine Black 4B is especially assisted by the presence of Oranit powder, Flerhenol M, Monopol soap, Neomerpin N, and Nekal A powder, the degree of assistance decreasing in the order named. A. J. HALL.

Wetting-out agents for [wool-]carbonising liquors. P. KRAIS (Textilber., 1926, **7**, 757).—The rate of sinking of woollen fabric in a carbonising liquor

(sulphuric acid of d 1·03) at 20° is greatly increased by the addition of but ½% of Flerhenol PF, Neomerpin, or Leonil, the beneficial effect of these wetting-out agents decreasing in the order named. A. J. Hall.

Dyeing leather. Lamb.—See XV.

Patents.

Dyeing cellulose acetate silk. R. Metzger, Assr. to I. G. Farbenind. A.-G. (U.S.P. 1,610,961, 14.12.26. Appl., 7.7.25. Conv., 11.7.24).—Compounds insoluble in water can be employed to dye cellulose acetate silk, either directly or by development on the fibre, if they are used in an aqueous colloidal suspension containing a compound which catalyses the hydrolysis of fats and oils, *e.g.*, a naphthenic acid, or Twitchell's reagent. Applied in this way 3-aminobenzanthrone gives strong yellow shades on cellulose acetate silk at 60—70°. T. S. Wheeler.

Dyeing or printing acetate silk. I. G. Farbenind. A.-G., Assees. of O. Spengler, P. Virck, and R. Weidenhagen (U.S.P. 1,615,242, 25.1.27. Appl., 7.12.25. Conv., 3.12.24; cf. F.P. 383,636; B., 1908, 332).—Cellulose acetate silk is treated in presence of ammonia and an oxidising agent (*e.g.*, hydrogen peroxide solution) with an arylamine (*e.g.*, *p*-aminophenol), which forms a coloured compound under such conditions, and is dyed fast tints without deterioration of the fibre. T. S. Wheeler.

Wetting-out agent and solvent. I. G. Farbenind. A.-G., Assees. of A. Knorr (G.P. 431,249, 24.12.24).—1 : 4-Dioxan, $O\langle\begin{smallmatrix}CH_2\cdot CH_2\\CH_2\cdot CH_2\end{smallmatrix}\rangle O$, m.p. 9°, b.p. 100°, and its derivatives are satisfactory organic solvents and wetting-out agents for use in dyeing, printing, wool-scouring, and in the spinning of artificial silk, varnish, leather-dressing, and rubber industries. A. J. Hall.

Production of a smooth, dull surface in dyed textile fabrics. G. A. Sallmann (E.P. 262,783, 7.12.26. Conv., 8.12.25).—The dry material, intended for use as leather substitute, is passed through rubber pressure rollers. B. P. Ridge.

Processes of bleaching and dyeing furs. F. B. Dehn. From Stein Fur Dyeing Co., Inc. (E.P. 265,324, 25.11.25).—See U.S.P. 1,564,378; B., 1926, 189.

Dyeing or colouring of products made with cellulose acetate. G. H. Ellis, Assr. to American Cellulose and Chemical Manuf. Co., Ltd. (U.S.P. 1,618,413—4, 22.2.27. Appl., 25.9.23. Conv., 27.1.23).—See E.P. 219,349; B., 1924, 906.

Dyeing materials comprising cellulose acetate and products produced. G. H. Ellis, Assr. to American Cellulose and Chemical Manuf. Co., Ltd. (U.S.P. 1,618,415, 22.2.27. Appl., 2.2.25. Conv., 4.4.24).—See E.P. 237,943; B., 1925, 801.

Apparatus for dyeing or washing textile goods and the like. J. Schlumpf (E.P. 253,500, 18.5.26. Conv., 9.6.25).

Stabilisation of bleaching baths (E.P. 265,417).—See VII.

Wastes from silk dyeing. Snell and Bruce.—See XXIII.

Disposal of waste. Hadley.—See XXIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Difficulties associated with blende roasting in conjunction with sulphuric acid manufacture. R. J. Harvey and W. H. V. Nettle (Proc. Austral. Inst. Min. Met., 1926, [63], 47—55).—In the roasting of Australian blende concentrates trouble is usually experienced with the formation of heavy scales on the rabbles and in the gas offtake. The maximum sulphur dioxide content of the flue gas is 5·5%; if a higher content is aimed at the furnaces become overheated with the consequent production of much lead sulphate fume, a good proportion of which finds its way into the lead chambers and even beyond. The only way to overcome the difficulties associated with deposition of lead sulphate throughout the acid plant is to stop operations for 2 hrs. every week and clear out all gas flues, the Glover towers, and the fans; this results, however, in a high nitre consumption, bad denitration, and poor concentration of the acid produced. Lead sulphate deposition in the towers causes loss of nitrous fumes and of sulphuric acid by adsorption. The presence of fluorine in the concentrates results in serious corrosion of the lead linings of coolers and the curtains of towers and chambers especially at the air-acid line. A. R. Powell.

Manufacture of arsenic acid. E. A. Suverkrop (Chem. Met. Eng., 1927, **34**, 96).—Arsenious oxide is delivered by a mechanical feed with variable speed control into a ferrosilicon, steam-jacketed kettle fitted with slow-moving agitators and a bottom discharge for the finished product. With this apparatus batches of over 1 ton are converted in 8—10 hrs. into nearly 3 tons of 75% arsenic acid, with recovery of 95% of the nitric acid used. Foaming of the hot batch has caused trouble, particularly when low-grade arsenious oxide is used, or if the feed is too rapid, and to overcome this the bulk of the reaction is carried out in the pump-chamber below the kettle, and the bubbles of nitrous gases are released from a fountain discharge above the surface level in the kettle. W. G. Carey.

Iodometric analysis of peroxides. C. Wagner (Pharm. Ztg., 1927, **72**, 218—219).—The official German method for the determination of peroxides is unreliable owing to the small amount of acid employed and to insufficient shaking. The following method gives accurate results:—0·3 g. of the peroxide is washed into a flask with water, 5 c.c. of 15% hydrochloric acid are added together with 15 c.c. of 10% potassium iodide solution, and the mixture is shaken continuously until the peroxide has dissolved. After 30 min. the liberated iodine is titrated with 0·1N-sodium thiosulphate solution. Small amounts of acid and insufficient agitation favour the side reaction $H_2O_2 + I_2 = 2H^{\cdot} + O_2 + 2I'$, with consequent low results. E. H. Sharples.

Sea waters and the potash problem. III. Preliminary industrial attempts to apply the Niccoli process. E. Niccoli (Giorn. Chim. Ind. Appl., 1926,

8, 603—610. Cf. B., 1925, 755).—Experiments are described on the application of the author's method for extracting potassium and magnesium salts from sea water (cf. F.P. 594,904, E.P. 247,405, 261,991; B., 1926, 237; 1927, 75) to the mother-liquors of the salt lakes of Bu-Kammasch (Tripoli). The mother-liquors from the evaporation of sea water give yields of potassium and magnesium salts 45% greater than the waters of the salt lakes. The evaporation pans may be prepared from the clayey soil contiguous to the salt lakes, this soil being systematically washed to remove the contained salts prior to use. T. H. Pope.

Dehydration of borax. M. A. Rakusin and D. A. Brodski (Chem.-Ztg., 1927, **51**, 95—96).—Neither absolute nor 95% alcohol has any action on borax at atmospheric temperature. With warming, 6·7 mols. of water are removed by alcohol of 95% strength or above, the loss decreasing with dilution of the alcohol, and reaching zero at 40% strength. The partly dehydrated mass is stable in the air. This process is, nevertheless, of only theoretical interest. Borax loses 5 mols. of water when kept over sulphuric acid in a desiccator or when heated on a water bath. By heating in an air oven to 140° 9 mols. of water are removed. The monohydrate could, therefore, be readily produced by heating with flue gases, and this is also stable under atmospheric conditions. The last molecule of water is only removed at a red heat, and as the anhydrous salt is hygroscopic no economic advantage would attend its preparation. C. Irwin.

By-products of the Chilean nitrate industry. J. B. Faust (Ind. Eng. Chem., 1927, **19**, 276—277).—In addition to the different grades of sodium nitrate and iodine some plants produce high potash nitrate and potassium perchlorate. A typical caliche contained 4·5% KNO_3 and 0·20% $KClO_4$. High-potash nitrate containing 30% KNO_3 is made by a crystallisation in stages or by refrigeration of the mother-liquor. Potassium perchlorate is precipitated where the quantity present warrants it by cooling the hot mother-liquor from the leaching tank to 50° and then pumping off the liquor for nitrate crystallisation. Borates are undesirable in sodium nitrate, and it might pay to precipitate them as manganese borate or zinc borate. C. Irwin.

Determination of perchlorate in Chile saltpetre with nitron. A. Vürtheim (Rec. trav. chim., 1927, **46**, 97—101).—Perchlorate may be determined in presence of nitrate by reducing the latter by the Devarda method, precipitating the perchlorate with nitron, filtering and weighing. The presence of iodate and borate is not detrimental. More consistent results are obtained than by the old method based on reduction of the perchlorate to chloride. H. F. Gillbe.

Some economic aspects of Texas potash. J. W. Turrentine (Ind. Eng. Chem., 1927, **19**, 271—274).—The potash deposits recently discovered in Texas comprise chiefly polyhalite (15% K_2O). Under existing conditions the only procedure likely to lead to profitable working would be mining and refining at the mine to anhydrous potassium magnesium sulphate (30% K_2O). Leaching in the ground and pumping, or transport of solution by pipe line to the coast, are impracticable, whilst the local scarcity of water is a difficulty in the procedure recommended above. On the other hand, conditions favour solar evaporation. It is suggested that these deposits will probably prove of only limited importance. C. Irwin.

Partial pressures of carbon dioxide, ammonia, and water over the system water–ammonia–carbon dioxide–ammonium nitrate. K. G. Clark and H. J. Krase (Ind. Eng. Chem., 1927, **19**, 205—207).—A current of purified air was passed through six bubbling tubes containing solutions of ammonium carbonate and ammonium nitrate and the exit gases were analysed. The results are tabulated in detail, and show that at temperatures from 20° to 60° addition of ammonium nitrate increases the vapour pressure of carbon dioxide, whilst it has little effect on that of ammonia. That of water is, of course, decreased. A similar effect was shown by ammonium sulphate and ammonium chloride. The separation of ammonia and carbon dioxide by distillation from an aqueous solution will therefore be facilitated by the presence of these salts. C. Irwin.

Removal of carbon dioxide from gas mixtures intended for ammonia synthesis. H. J. Krase and H. C. Hetherington (Ind. Eng. Chem., 1927, **19**, 208—211).—In the water-gas method of preparing the gases for ammonia synthesis carbon dioxide is usually removed by scrubbing the gases compressed to 25—100 atm. with water. Recovered in this way, the carbon dioxide is not pure enough for use in the synthesis of carbamide. An experimental plant was constructed to scrub the gases with ammonia solution, to which ammonium nitrate had been added to assist regeneration (*vide supra*). The economical working of the scrubber depended on the efficiency of the regenerator which governed the volume of solution necessary. Under similar flow conditions an efficiency of 90% of carbon dioxide removal was obtained with a solution containing 22% of ammonium nitrate as against 48—50% with ammonium sulphate or water only. It is estimated that the cost of ammonia scrubbing is 5% less than that of high-pressure water scrubbing, to which the value of the carbon dioxide is to be added. Difficulty was experienced through corrosion of iron pipes and valves by the solution, and it was found that very concentrated solutions of ammonium nitrate were markedly reduced by iron. Aluminium, however, was unaffected. C. Irwin.

Solid carbon dioxide as a commercial refrigerant. D. H. Killeffer (Ind. Eng. Chem., 1927, **19**, 192—195).—Solid carbon dioxide is manufactured from the gases of combustion of coke burnt under a special steam boiler, the whole of the power generated being required for compression. The exit gases containing 17—18% CO_2 pass through towers where they are washed with water, then through to a fan to coke-packed absorption towers fed with a 10% sodium carbonate solution. The latter is stripped by heating at 115°, yielding a gas of 99·9% purity. This is cooled and liquefied at a pressure of 1100 lb./sq. in. The liquid carbon dioxide is allowed to expand into the inner chamber of a jacketed vessel covered by a filter cloth to retain the "snow." The cold gas passes around the jacket and a yield of over 33% of solid is obtained. This is compressed into blocks,

which are packed in ordinary packing cases of wood. Owing to the elimination of the cylinders necessary for liquid carbon dioxide the solid can be profitably sold at one half the price of the latter. Owing to the insulating effect of the cold gas produced it is remarkably stable, and the absence of any liquid product is convenient. It is economically applicable for use in refrigerating cars making long journeys, 1 pt. replacing 15 pts. of water-ice and 3 pts. of salt. C. Irwin.

Absorption of nitrogen oxides from ammonia oxidation. C. Toniolo (Chem. Met. Eng., 1927, **34**, 92—95).—Cooling of the absorption towers in the manufacture of nitric acid is advisable to retard the decomposition of the concentrated acid by thermal and chemical action, such as the reduction by nitric oxide, which is much faster in strong than in weak acid, and also the mutual decomposition of nitrogen trioxide and nitric acid. Cooling also promotes polymerisation of nitrogen oxides, preventing the production of nitrogen trioxide, and it also accelerates the oxidation of nitric oxide. For efficient operation the oxidation of nitrogen should be effected to the maximum extent, and the heat evolved should be removed by cooling before starting the absorption in water. Thorough contact of gas and liquid in the absorption tower is of the utmostimportance; the diameter of the towers should therefore be as small as possible, and they should not be filled with symmetrical fillers, but with irregularly shaped materials to compel the gases and liquids to traverse irregular and constantly changing paths. W. G. Carey.

Determination on the spot of traces of nitrogen peroxide in air. E. Kohn-Abrest (Compt. rend., 1927, **184**, 482—484).—A method for the determination of nitrogen peroxide in air is described in which the sample is shaken with $0{\cdot}1N$-sodium hydroxide solution, and the nitrite produced according to the equation $2NO_2 + 2NaOH = NaNO_3 + NaNO_2 + H_2O$ is titrated with $0{\cdot}1N$-potassium permanganate solution. Precautions must be taken in the presence of ozone, sulphur dioxide, and hydrogen sulphide. No nitrogen peroxide has been detected in the atmosphere of a room in which an ultra-violet lamp had been working for 30 min.

J. Grant.

Patents.

Production of titanic acid unaffected by light. Deutsche Gasglühlicht- Auer- Ges.m.b.H. (E.P. 257,259, 13.8.26. Conv., 20.8.25).—White titanic acid, unaffected by light, is produced by the hydrolysis under pressure of titanium sulphate solution containing such a small amount of tervalent titanium that, after hydrolysis, the solution contains a trace only of tervalent titanium, or none at all. W. G. Carey.

Method and apparatus for the concentration or purification of caustic soda or other suitable material. C. F. Hammond and W. Shackleton (E.P. 265,252, 5.9.25).—The material is conducted in a circuitous course over the surface of a molten mass of lead or suitable alloy, which is heated in a specially devised receptacle by a submerged flame burner, the products of combustion being withdrawn from a sealed and enclosed space away from the material. The hot liquid or molten mass may be circulated by the air-lift action of the combustion gases. W. G. Carey.

Oxidation of ammonia. F. G. Liljenroth (F.P. 610,010 and 610,160, [a, b], 26 and 27.1.26. Conv. [a, b], 29 and 30.1.25).—(a) Ammonia is oxidised in stages, the gases being cooled between the stages. (b) A portion of the mixed gases obtained by the oxidation of ammonia, consisting principally of nitric oxide, steam, and oxygen, is cooled, mixed with more ammonia and oxygen, and returned to the process.

L. A. Coles.

Separating solid salts of ammonium and of the alkalis and alkaline earths. O. Y. Imray. From Farbwerke vorm. Meister, Lucius, & Brüning (E.P. 242,975, 4.11.25).—Solid ammonium salts made by double decomposition from alkali salts or alkaline-earth salts are separated therefrom in an elutriating apparatus, which is fed with a mother-liquor of high sp. gr.

W. G. Carey.

Manufacture of alkali cyanides. S. Coulier (E.P. 265,305, 7.11.25).—Organic matter rich in nitrogen, such as animal waste, liquors from sugar works, wool washing, sewerages, etc., is distilled with an alkali compound and the nitrogenous gases (nitrogen, ammonia, amines, etc.) freed during the distillation are caused to act at a high temperature on the residue of the distillate containing carbon and alkali, which prior to this has been treated in a closed vessel under pressure of an inert gas to fix as cyanide the residual nitrogen therein. If the nitrogenous matter contains sulphur, a desulphurising agent, *e.g.*, alkaline-earth oxides or carbonates, a metal, or a metallic oxide, is added before the distillation. W. G. Carey.

Manufacture of alkali metal cyanides. Deutsche Gold- u. Silber-Scheideanstalt vorm. Roessler, Assees. of H. Freudenberg (E.P. 265,639, 15.2.26).—In the manufacture of alkali cyanides from alkali metals, carbon, and ammonia, the hydrogen liberated is purified by washing with water in coke-scrubbing towers, and after drying is mixed with nitrogen for the catalytic synthesis of ammonia, which is then introduced into the cyanide process. W. G. Carey.

Preparing magnesium cyanide and product thereof. W. E. Olberg, Assr. to California Cyanide Co., Inc. (U.S.P. 1,609,038, 30.11.26. Appl., 22.5.26).—Magnesium nitrate, with or without prior solution in water, is treated with ammonia, and an excess of hydrogen cyanide, and the precipitate obtained is dried *in vacuo* at 40° for ½ hr. It has the formula $Mg(CN)_2,xNH_3$, and is of value as a fumigant, evolving hydrogen cyanide and ammonia in contact with air. T. S. Wheeler.

Producing [pure] heavy-metal cyanides [from crude cyanides]. K. F. Cooper, Assr. to American Cyanamid Co. (U.S.P. 1,614,523, 18.1.27. Appl., 13.5.22; cf. 1,282,395 and 1,359,257; B., 1919, 12 a; 1921, 9 a).—An aqueous solution of a crude commercial cyanide, *e.g.*, calcium cyanide, is filtered and treated with a salt of a heavy metal which does not precipitate calcium by double decomposition, *e.g.*, zinc chloride, in quantity sufficient to form a soluble double cyanide of the type $Ca(CN)_2,Zn(CN)_2$, a slight excess of zinc chloride being

added to precipitate any ferrocyanide and sulphide present. The solution is filtered, and is then treated with a further quantity of zinc chloride, when pure zinc cyanide is precipitated. Copper, silver, and cadmium cyanides are similarly obtained. T. S. WHEELER.

Recovery of sodium sesquicarbonate from brine. W. A. KUHNERT (U.S.P. 1,618,834, 22.2.27. Appl., 27.7.25).—Carbon dioxide is passed into brine at such a rate that less than 30 lb./hr. per 1000 gals. of brine are absorbed, and the precipitated sodium sesquicarbonate is then separated. W. G. CAREY.

Recovery of bromine [from brine]. H. H. DOW and E. O. BARSTOW, Assrs. to DOW CHEMICAL Co. (U.S.P. 1,614,663, 18.1.27. Appl., 20.9.24; cf. 1,323,173; B., 1920, 518 A).—Warm brine containing a small proportion of bromine as bromide is chemically treated to liberate the bromine, which is removed in a tower by a countercurrent of air, which is then freed from bromine with sodium hydroxide solution, and returned to the brine-treating tower. The spent warm brine is passed down a second tower, against an up-current of air, which is heated by the brine, and is then employed in a third tower to preheat fresh brine, and returned to be reheated by spent brine, thus effecting a heat economy. T. S. WHEELER.

Rendering table salt non-hygroscopic. "A.P.I.C.E." SOC. ANON. PRODOTTI ITALIANA CHIMICI ESTRATTIVI (F.P. 609,404, 13.1.26. Conv., 19.1.25).—Sodium chloride, or a solution containing it, is treated with sodium, potassium, or lithium carbonate. L. A. COLES.

Manufacture of hypochlorites. M. C. TAYLOR, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,609,328, 7.12.26. Appl., 29.10.24; cf. E.P. 195,366; U.S.P. 1,481,040; B., 1924, 173, 334).—Chlorine is passed into an agitated, aqueous suspension of *p*-nitroacetanilide, or similar amine, and calcium carbonate, when calcium chloride passes into solution, and *p*-nitroacetochloroanilide is precipitated, and recovered by filtration. On treatment with ice and calcium hydroxide, it yields insoluble *p*-nitroacetanilide and soluble calcium hypochlorite, which is recovered in a substantially pure condition by evaporation of the solution *in vacuo*. Several modifications of the process are possible, *e.g.*, the amine is dissolved in a solvent, such as carbon tetrachloride, which is immiscible with water, and which also dissolves the *N*-chloroamine formed. T. S. WHEELER.

Stabilisation of bleaching baths [containing peroxides or persalts]. T. BENCKISER, A. REIMANN, A. REIMANN, JUN., and F. DRAISBACH (E.P. 265,417, 7.4.26).—Solutions of hydrogen peroxide, perborates, or other persalts are stabilised by the addition of an acid alkali-metal pyrophosphate, the resulting degree of stabilisation being much greater than that obtained by the similar use of a neutral pyrophosphate (cf. E.P. 23,676; B., 1910, 152). A solution consisting of 0·4 g. of an acid pyrophosphate, 2 g. of a perborate, and 150 c.c. of water contains 92% of its original active oxygen after heating for 4 hrs. A. J. HALL.

Manufacture of zirconium compounds. C. J. KINZIE, Assr. to TITANIUM ALLOY MANUF. Co. (U.S.P. 1,618,286—7, 22.2.27. Appl., [A] 22.4.25, [B] 15.7.25).—(A) To obtain an acid-soluble zirconium compound, the ore is fused, with partial expulsion of silica, and is then heated with sulphuric acid at 350—450°. (B) Basic potassium zirconium sulphate is made by adding an alkali to a solution of normal zirconium sulphate, treating the mixture with potassium sulphate, and separating the precipitate formed. W. G. CAREY.

Manufacture of new complex auro-sodium thiosulphate solutions. L. CASSELLA & Co. (E.P. 246,809, 17.12.25. Conv., 27.1.25)—Solutions which are stable, and which can be sterilised by heat, are produced by allowing sodium thiosulphate to act upon auro-sodium thiosulphate, either by dissolving a mixture of the two components, or by dissolving them separately and mixing the components in solution. W. G. CAREY.

Conversion of hydrogen sulphide into sulphurous acid. F. SIEMENS A.-G., G.M.B.H., Assees. of H. BÄHR (F.P. 609,931, 25.1.26. Conv., 6.8.25).—Hydrogen sulphide is brought in contact at 280—320° with a catalyst containing a metal capable of combining with sulphur, *e.g.*, copper, nickel, or iron, and an oxygen carrier, *e.g.*, vanadium, chromium, or molybdenum. L. A. COLES.

Direct absorption of nitric oxide. T. SCHLOESING (F.P. 609,264, 3.4.25).—Nitrous gases are brought in contact with basic material insoluble or slightly soluble in water, *e.g.*, calcium carbonate, in the presence of water. L. A. COLES.

Deposition of boron in coherent form. W. J. TENNANT. From N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 264,953, 9.11.25).—The apparatus consists of a glass tube provided with leading-in wires for an electric current, connected by the tungsten or other metal wire to be coated. Two bulbs are sealed to the bottom of the tube; in one, which is kept at room temperature, is placed a halide of boron, preferably the bromide; the other is cooled by means of liquid air. The boron halide volatilises and, as it comes into contact with the wire, which is electrically heated at 1400°, is dissociated, boron being deposited on the wire, and the halogen set free, uniting with mercury contained in the bottom of the tube. Any undissociated boron halide condenses in the cooled bulb. When all the boron halide has volatilised from the one bulb, the functions of the two bulbs may be interchanged. The atmosphere should consist exclusively of boron halide, as some atmospheres, *e.g.*, hydrogen or nitrogen, have a detrimental effect on the deposition. M. E. NOTTAGE.

Catalytic apparatus [for synthesis of ammonia]. F. H. BRAMWELL, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,618,265, 22.2.27. Appl., 1.5.26. Conv., 4.5.25).—See E.P. 241,817; B., 1926, 12.

Extraction of ammonium aluminium sulphate from aluminium sulphate solutions containing ferric compounds. R. GANSSEN (U.S.P. 1,619,666, 1.3.27. Appl., 14.2.21. Conv., 21.9.17. Renewed 11.12.26).—See E.P. 132,510; B., 1921, 79 A.

VIII.—GLASS; CERAMICS.

Apparatus for measuring the turbidity of opalescent glass. W. EWALD (Z. angew. Chem., 1927, **40**, [Chem. App. u. Maschinenw.], 32).—A photometer is

described in which the proportion of light that passes through the glass (*a*) directly, (*b*) in a diffused condition and (*c*) the proportion that is scattered by diffuse reflexion may be determined. Parallel beams of light from a common source are passed through two tubes of colloidal thorium oxide solution arranged at an angle to each other, and the issuing light is reflected from mirrors on to a photometer head on which the light passed by two glasses, one a standard and the other that under test, placed one at the beginning of each tube, is compared. The light diffused by passing through the glass is compared by means of a second photometer placed between the two tubes, so that light diffused from each glass at an angle of 40° with the incident beam is measured. Reflected diffused light is measured in a similar way when the glasses are placed at the other end of the thorium oxide tubes. A. R. POWELL.

Causes and prevention of [ceramic] scumming and efflorescence. L. P. COLLIN (Dept. Mines, Canada, 1925, Rep. No. 672, 9—16).—Scumming is defined as a deposit of foreign matter formed on the surface of a ceramic body during the process of manufacture; efflorescence as a deposit on finished ware due to exposure to weather. Scumming may be caused by soluble salts in the raw material or in the tempering water, and the effect of these may be removed by weathering if pyrites is not present, by the addition of barium salts, or by rapid drying. Other causes are the formation of sulphates in the dryer and subsequent deposition in the kiln, or the formation of sulphates in the kiln itself, which causes may be counteracted by the elimination of sulphur gases from the dryer by the use of clean, dry, non-sulphurous fuels, by the addition of barium salts, and by alternate reducing and oxidising conditions in the kiln. The chief causes of efflorescence are the presence of soluble salts, particularly the sulphates of magnesium, sodium, and potassium, in the fired ware; by the absorption of such salts from mortar, stone copings, or the ground; or soluble salts formed by weathering. Efflorescence may be checked by hard firing and by firing in a reducing atmosphere or under alternate oxidising and reducing conditions. F. SALT.

Texture of ceramic materials. J. F. McMAHON (Dept. Mines, Canada, 1925, Rep. No. 672, 17—22).—Texture is determined by the shape and size of the grain of the materials of which a body is composed. Fine, angular grains increase the plasticity and tend to produce vitrification during firing. Coarse, round grains, on the other hand, are advantageous for drying and in the water-smoking and oxidation periods of firing. The presence of large grains reduces the strength both of the fired and unfired product; a fine-grained product, being close-textured and having great bonding strength, is more resistant to abrasion, corrosive action, and scumming. Resistance to abrupt changes of temperature (spalling), however, is enhanced by the presence of coarse particles. The grading or proportioning of grains of different sizes is an important factor.
F. SALT.

Anhydrous silicas containing clays. Composition of ochres. A. P. BIGOT (Compt. rend., 1927, **184**, 381—383).—A number of siliceous rocks have been ground, and the portion passing through a No. 300 sieve was treated by levigation, or the amorphous silica dissolved out by means of sodium hydroxide solution. Clays varying in amount from 9·4 to 28·0% have been found, often associated with chalcedonite, in the unattacked residue. Globular silica and silica obtained from ochre, diatoms, and infusoria also retain clay, the silica being anhydrous and deposited in an agglomerated form in the clay. Ochres are a mixture of globular silica, chalcedonite, clay, and iron hydroxide.
J. GRANT.

PATENTS.

[Translucent] glass composition. W. C. TAYLOR, Assr. to CORNING GLASS WORKS (U.S.P. 1,615,247, 25.1.27. Appl., 2.11.22).—Improved translucent, but not transparent, glass is obtained by fusing a batch containing at least 70% of silica in the form of sand ground finer than 100 mesh, at least 3% of boric oxide, an alkaline content less than that of the boric oxide, not more than traces of lead oxide, and 1—5% of an alkaline chloride or bromide. T. S. WHEELER.

Cast refractory article. Cast refractory product. G. S. FULCHER, Assr. to CORNING GLASS WORKS (U.S.P. 1,615,750—1, 25.1.27. Appl., [A], 31.7.25; [B], 27.7.26. Conv., [B] 25.6.26).—(A) A mixture of refractory materials, comprising (B) about 30—40% of silica, 50—30% of alumina, 10—30% of zirconia, and an alkaline content of less than 15%, is fused in an electric furnace, and run into a mould of glass sand, or like refractory material, mixed with a binder, *e.g.*, linseed oil. When the outer surface has set, the casting is removed from the mould, and annealed by slow cooling in an oven, or immersed in ground refractory material. The product is non-porous, and resistant to the action of molten glass. T. S. WHEELER.

Enamelling metal articles and the like. C. NOËL (F.P. 609,844, 5.5.25).—The articles are treated with a current of air while the portion to be enamelled is raised gradually to a suitable temperature. L. A. COLES.

Refractory articles or castings. R. HADDAN. From CORNING GLASS WORKS (E.P. 265,847, 25.6.26).—See U.S.P. 1,615,750—1; preceding.

Glass-annealing lehrs. HARTFORD-EMPIRE CO., Assees. of H. W. INGLE (E.P. 250,200, 13.3.26. Conv., 31.3.25).

Shock-resistant glass. W. R. INNES (E.P. 265,319, 21.11.25).

IX.—BUILDING MATERIALS.

Portland cement. Improvements in manufacturing processes and quality. H. J. HAWKES (Proc. Austral. Inst. Min. Met., 1926, [63], 57—61).—After reviewing recent improvements in cement manufacture in this country and in France, comparative tests of the hardness and setting properties of Australian quick-hardening cement, "ferrocrete," and a slow-hardening Portland cement are described. The first-named contains 6·6% Al_2O_3, and is supplied ground so that 95% passes a 180-mesh sieve; its tensile strength 7 days after mixing with water is slightly greater than that of ferrocrete, but its compression strength is only

two-thirds as great. Concrete roads made of this cement are available for use within 3 days.

A. R. POWELL.

PATENTS.

Obtaining heavy spar and a paint or a material for cement manufacture. K. EBERS (E.P. 246,498, 22.1.26. Conv., 22.1.25).—A mineral containing heavy spar and calcium carbonate is calcined and the product slaked with water. The heavy spar is separated from the slaked lime and other impurities by decantation with water, treatment with crude hydrochloric acid being given if necessary. The separated lime and impurities can be used as a paint or as a raw material for cement manufacture. B. W. CLARKE.

Manufacture of alumina-containing cements. G. HERTZKA (E.P. 265,494, 5.10.26).—Raw materials for aluminous cement manufacture in a comparatively coarse state of division (leaving a residue of 1% on a sieve 900 meshes/cm.^{2}) are shaped into briquettes etc., which are preheated for 24 hrs., and then maintained for 30 hrs. in a state of rest at a temperature between 1150° and the m.p. of the mixed materials, the clinker produced being cooled over a period of 30 hrs. before grinding.

B. W. CLARKE.

Cement mixture. F. H. CHRISTIANSEN (U.S.P. 1,618,512, 22.2.27. Appl., 26.4.22. Renewed 22.10.24). —Cement and sawdust are mixed with an aqueous solution of calcium chloride (*d* 1·0016).

B. W. CLARKE.

Pitch cement. CONTINENTALE PRODORIT A.-G. (G.P. 434,779, 15.1.25).—A product resistant to chemical action and having high mechanical strength consists of hard pitch containing as little free carbon as possible and having a softening point (Krämer–Sarnow) of at least 110—160°, mixed with sand or finely-divided rocky material. L. A. COLES.

Manufacture of refractory bricks from chamotte and casting clay slips. SCHEIDHAUER & GIESSING A.-G. (E.P. 253,947, 22.6.26. Conv., 22.6.25).—Binding clay is liquefied in a known manner, *e.g.*, by the addition of water and an electrolyte, such as alkali, and the concentrated, preferably alkaline, clay slip produced is mixed with sufficient grog to produce a non-castable mass, which is moulded by beating or under pressure to ensure close contact of the grains, dried, and then burnt, producing articles with a high binding strength and resistance to pressure. Blocks of low plasticity are obtained by diluting the clay slip with water, mixing this with the grog, moulding as above, drying, and burning; if grog previously burnt at a high temperature is used, the dried, moulded articles can be used without burning. B. W. CLARKE.

[Agents for] emulsification. J. T. JONES (E.P. 264,955, 10.11.25. Cf. 222,602; B., 1924, 987).—The emulsifying agent is obtained by treating aqueous dispersions of fish or animal proteins with dilute caustic alkali at a temperature below 100°, sufficient to maintain the whole in a fluid condition. By its aid emulsions of bitumen or tar, with or without the addition of mineral oils, adapted for the treatment of roads may be obtained.

F. R. ENNOS.

Manufacture of Portland cement. C. PONTOPPIDAN (U.S.P. 1,618,295, 22.2.27. Appl., 3.4.26).—See E.P. 260,447; B., 1927, 13.

Mixing machine for concrete, tar macadam, and the like. L. J. B. BLAKE (E.P. 265,304, 7.11.25 and 20.7.26).

Drying machines [for tar macadam] (E.P. 264,921—2 and 264,931).—See I.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Effect of prolonged overheating on the structure and properties of cast iron. F. MEYER (Stahl u. Eisen, 1927, 47, 294—297).—A series of 77 castings of iron of different compositions melted under various conditions has confirmed Piwowarsky's observations on the effect of prolonged over-heating on the structure (cf. B., 1925, 805). The proportion of graphite to total carbon increases with the temperature of heating up to about 1420—1475° according to the silicon content, then decreases. The greater the overheating the finer are the graphite particles in the cast or slowly cooled metal and the more regular is their dispersion. A. R. POWELL.

Physical structure of cast iron and its relation to cupola practice. E. HOWELL (Proc. Austral. Inst. Min. Met., 1926, [63], 11—16).—To obtain good castings free from segregation and sponginess the following composition of the iron is recommended: 2·0—2·5% Si, 0·03% S, 0·15—0·25% P, 1·0—1·5% Mn, remainder Fe. For melting this metal at the rate of 2·5—3·0 tons/hr. a suitable cupola would have an external diameter of 3 ft. 6 in., an internal diameter of 2 ft. 6 in., with a height of 12 ft. to the charging door. The main tuyères would be 4·5 in. diameter, fed with an 8 in. blast pipe from a No. 2 Roots blower at 4—5 oz. air pressure. A suitable fire-brick lining would consist of 74—75% SiO_2, 0·9—1·7% Fe_2O_3, 22—24% Al_2O_3, and less than 0·6% $CaO+MgO$. Using a coal with fairly low ash content a high initial tapping temperature should be readily obtained even when casting a semi-steel. Characteristic photomicrographs are shown of the structure of cast iron obtained under different casting conditions.

A. R. POWELL.

Penetration of mild steel by brazing solder and other metals. R. GENDERS (Inst. Metals, March, 1927, advance copy, 7 pp.).—The cracking of mild steel under stress when heated and wetted by brazing solder has been studied, and is due to intercrystalline penetration by brass. Similar penetration is found with molten copper, but zinc, tin, or lead-tin solders have little action. When a rod coated with molten brass is bent, the penetration takes place rapidly in the part which is in tension, but not in that in compression. This kind of fracture under stress of iron or other metals exposed to molten alloys or corrosive agents is due to penetration, and not to previous intercrystalline weakness. The possible effect of an unknown accelerating factor is discussed. W. HUME-ROTHERY.

Manufacture and properties of hair-springs. H. MOORE and S. BECKINSALE (Inst. Metals, March, 1927, advance copy, 9 pp.).—Different types of hair-

springs and control springs are enumerated, and their functions and essential properties are described. Both ferrous and non-ferrous alloys are used for the manufacture of such springs, and methods of hardening by quenching and tempering and by cold-working are discussed. Some form of hardening is necessary in all cases to raise the elastic range to the required degree, but all hardening processes are liable to produce material with imperfect elastic properties. Carbon steel of high quality containing 1—1·3% C is most frequently employed, and is suitably hardened by quenching in oil from 750° and tempering at 300—350°. Liability to corrosion is the chief disadvantage of this material, but certain ferrous alloys are now used which are highly resistant to corrosion. Chief amongst these is Elinoar, which contains about 12% Cr and 36% Ni. This can only be hardened by cold-working, and hair-springs made from it are tempered by heating at 350—600°. The possibilities of non-ferrous alloys are indicated, and their merits are compared with those of steel and ferrous alloys. The manufacture and properties of phosphor-bronze hair-springs are detailed, and alloys used for hair-springs where a low electrical resistance is required are discussed. M. Cook.

Rapid determination of silicon in steel and iron. J. Verfürth (Chem.-Ztg., 1927, **51**, 122—123).—This method can be carried through in 50—60 min. 0·5—3·0 g. of the sample are dissolved in 20—30 c.c. of brominated hydrochloric acid (5 pts. of hydrochloric acid *d* 1·19, 5 pts. of water, 1 pt. of bromine) and evaporated to dryness. The residue is dissolved in 10—15 c.c. of hydrochloric acid (*d* 1·19), diluted, and filtered through a suitable filter paper. Any silica adhering to the sides of the vessel used is readily removed. The filter paper is burnt in a porcelain crucible and ignited to constant weight. Ignition is rapid, and the silica is in such a condition that no fluorine treatment is necessary. The error will not exceed 0·02%. For high-silicon irons of 5% Si and above, a platinum crucible and fluorine treatment should be used, and silica must be recovered from the filtrate, as with this material graphite is not completely destroyed by the bromine treatment. For ordinary irons the two errors, however, cancel each other with fair exactness. Details of a number of tests are given. C. Irwin.

Hot-shortness of brass. R. Mailänder (Z. Metallk. 1927, **19**, 44—51).—The brittleness of brass at high temperatures has been examined by dropping a weighted, wedge-shaped bar on to a test piece of the metal supported on two knife edges and observing the height of fall required to cause fractures to appear. Annealed brass (72% Cu) may be bent in this way to an angle of 120° without fracture at temperatures below 500°, whereas in the cold-rolled or drawn state it commences to fracture above 200°. The height of fall (H) necessary to cause rupture decreases with the time of heating the specimen to a minimum (H_m) at the critical period (P_K), and then increases. Both H_m and P_K decrease with the temperature slowly at first, then rapidly above 400°; P_K also decreases linearly with the hardness of the specimen, the slope of the curve being greater the lower the temperature of the test, whereas H_m first decreases to a minimum, and then increases with the hardness. The angle to which the blow bends the specimen at the breaking point decreases with the value of H_m. With a constant value of H the angle remains constant for annealing temperatures below 300°, but an extremely large increase occurs on annealing just above 300°. That the brittleness of this brass is a function of the temperature is shown by the fact that after quenching from 500° the metal behaves normally in the test. Addition of small quantities of lead to brass intensifies the brittle behaviour of the metal at high temperatures. These results indicate that sudden heating of severely cold-rolled brass may lead to the development of internal cracks. A. R. Powell.

Magnesium-rich magnesium-copper alloys. M. Hansen (Inst. Metals, March, 1927, advance copy, 8 pp.).—Tensile and hardness tests have been carried out on magnesium-copper alloys containing up to 15% Cu. Elongation and reduction of area are higher in the quenched than in the slowly-cooled state, but the ultimate strength and hardness are little affected. By the micro-examination of slowly-cooled and quenched specimens, it is shown that the solubility of copper in magnesium increases from about 0·1% at room temperature to about 0·4—0·5% at 485°. The fact that no change in hardness is found on quenching is due to the atomic mobility being sufficiently great to enable the precipitation of the compound Mg_2Cu to take place during the quenching operation. W. Hume-Rothery.

Application of strain methods to the investigation of the structure of eutectic alloys. F. Hargreaves (Inst. Metals, March, 1927, advance copy, 3 pp.).—Suitable methods of straining yield markings in eutectic alloys due to slip which are helpful in determining the crystal unit in such alloys. In the tin–lead eutectic the crystal unit may be a eutectic "colony" or a unit containing only the two constituents in laminated form. The eutectic alloy and alloys containing excess of tin deform in a manner similar to that metal, indicating that tin determines the orientation. Tin also determines the orientation of the tin–zinc eutectic. In the copper–silver eutectic one of the structural units is the eutectic "colony." M. Cook.

Precipitation of zirconium with picric acid. M. Speter (Metallbörse, 1926, **16**, 1449—1450; Chem. Zentr., 1926, II, 2740—2741).—Zirconium picrate is precipitated quantitatively by the addition of picric acid to neutral or nearly neutral solutions of zirconium compounds. Filtration of the precipitate is facilitated by boiling the solution. Cautious ignition of the precipitate is necessary as it is likely to explode, but the tendency is diminished by moistening it with sulphuric acid prior to ignition. Titanium and thallium salts are not precipitated by picric acid, and the method is applicable for separating zirconium from mixtures of it with iron or thallium. L. A. Coles.

Electric furnaces in non-ferrous metallurgy. D. F. Campbell (Inst. Metals, March, 1927, advance copy, 11 pp.).—The Ajax–Wyatt and Ajax–Northrup induction melting furnaces are described, and details of the costs of operation are given with special reference to non-ferrous metallurgy. W. Hume-Rothery.

PATENTS.

Making molybdenum steel. F. M. BECKET, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,615,501, 25.1.27. Appl., 8.10.25).—A molybdenum sulphide concentrate is roasted in presence of air with lime, or similar alkaline or alkaline-earth compound, and added to molten steel, when most of the molybdenum present is reduced and alloyed with the steel, in which there is no increase in sulphur content. T. S. WHEELER.

Preventing the rusting of iron and steel in contact with water and hydrocarbons. L. H. J. M. MARTINEAU (F.P. 610,686, 6.2.26).—Water or hydrocarbons flow over lime before coming in contact with iron or steel, or iron vessels for containing water or hydrocarbons are coated with lime. L. A. COLES.

Non-corrosive steel. A. WEBER, Assee. of F. SOMMER and F. RAPATZ (G.P. 431,030, 2.10.21).—Non-rusting steel which is not attacked by salt solution or by concentrated sulphuric acid, and only slightly by dilute sulphuric or hydrochloric acid, contains 0·1—1% C, 1—5% Si, 8—20% Cr, and 1—5% Cu.
L. A. COLES.

Utilisation of ferrosilicon masses containing valuable metals. T. GOLDSCHMIDT A.-G. (E.P. 251,268, 16.4.26. Conv., 23.4.25).—Small amounts of valuable metals, *e.g.*, tin, may be recovered from ferrosilicon by the addition to the molten masses of such metals, alloys, or compounds reducible to metals as do not alloy with the iron or silicon, but which alloy easily with the valuable metals; *e.g.*, lead, bismuth, cadmium, or thallium, may be added separately or together. M. E. NOTTAGE.

Method of annealing copper articles. S. ST. P. MEEK (U.S.P. 1,617,859, 15.2.27. Appl., 18.1.26).—The article is packed in powdered metallic copper, placed in a suitable vessel, and the entire container heated.
M. E. NOTTAGE.

Extraction of lead from materials containing it or its compounds. S. C. SMITH (E.P. 264,569, 21.7.25). —The finely-ground ore is either treated with strong hydrochloric acid in excess at ordinary temperatures, whereby the lead is converted into lead chloride while other metallic sulphides present, such as zinc sulphide, remain unattacked; or the ore may be treated with hydrochloric acid of weak or moderate strength at ordinary temperatures, the liquor separated from the residual solid matter, and the latter treated with a more concentrated solution of hydrochloric acid. This process may be repeated any number of times, strong hydrochloric acid in excess being used for the final extraction. The weak acid liquor resulting from the attack of strong acid on the ore may be used to chloridise more of the sulphide after the latter has been treated with an oxidising agent consisting of liquor containing iron salts which has been produced in a previous operation, and oxidised. The operation may be carried out in a rotating barrel, the ore being made to enter at one end and the fresh liquor at the other. The hydrogen sulphide liberated should be removed from the solution either mechanically or by the use of an oxidising agent, such as sulphur dioxide, in order that the reaction may proceed to completion. The treated ore is washed with cold water and the lead chloride extracted from it either by leaching with hot water or brine, or by flotation. Lead ores which contain silver may be treated in a similar way with hydrochloric acid, preferably at 40°, or other reagent, either before or after removal of lead chloride, the silver being finally precipitated from the solution as iodide and recovered. The ore may be subjected to a preliminary treatment with sulphuric acid in order to remove acid-consuming materials. M. E. NOTTAGE.

Extraction of gold from dilute solutions. W. O. SNELLING (U.S.P. 1,617,353, 15.2.27. Appl., 12.2.25).—The dilute solution of gold is passed through a porous mass of amorphous precipitated tellurium.
M. E. NOTTAGE.

Method and means for producing galvanic chromium coatings on hollow articles, especially on narrow tubes. SIEMENS & HALSKE A.-G. (E.P. 259,900, 21.1.26. Conv., 17.10.25).—The hollow article to be coated forms the cathode in a vessel containing the electrolyte. The anode consists of two parts which, by means of suspension wires passing over rollers, can move upwards relatively to the interior and exterior surfaces, respectively, of the cathode at a uniform velocity, such that the resulting coating is of the desired thickness at every part. The length of the anode should be about one-tenth that of the cathode. The electrolyte is continuously introduced into the vessel at such a rate that it is always at a constant height above the upper edge of the movable anode. The suspension wires serve as current conductors, and the electrodes are suitably insulated from each other inside the vessel. A very adherent coating is produced.
M. E. NOTTAGE.

Electrolytic separation of metallic chromium. R. APPEL (E.P. 265,833, 17.5.26. Addn. to 259,118; B., 1926, 985).—In the electrolytic separation of chromium carried out in accordance with the prior patent there is added to the dilute solution of chromic acid or chromates used as bath liquid 0·05—0·1% of sodium sulphate, potassium sulphate, or bisulphate, magnesium sulphate, or neutral or acid phosphates, or electrolytes containing inorganic or organic acids.
J. S. G. THOMAS.

Method and apparatus for removing hydrogen from the surface of metal objects to be coated by electrolysis. L. J. J. VAN DER HOORN (E.P. 265,047, 12.5.26).—The layer of hydrogen may be removed by the use of gas supply-pipes provided with horizontal perforated arms extending radially from the centre of an annular tank and capable of being rotated about the axis of the latter. The gas (*e.g.*, air), supplied to the pipes through a hollow shaft in the axis of the apparatus, rises periodically from below and intercepts the objects to be treated. M. E. NOTTAGE.

Method of decarbonising ferrochrome. W. B. D. PENNIMAN and E. J. SHACKELFORD (E.P. 262,795, 3.9.25. Conv., 11.12.24).—See U.S.P. 1,527,088; B., 1925, 362.

Treatment of ores and metallurgical products. F. JOHANNSEN, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,618,204, 22.2.27. Appl., 6.12.24. Conv., 7.12.23).—See E.P. 225,842; B., 1925, 597.

Separation of minerals by froth-flotation. L. A. WOOD and MINERALS SEPARATION, LTD. From C. P.

Lewis (E.P. 262,492, 12.8.25).—See U.S.P. 1,610,298; B., 1927, 168.

Firearm and alloy for making same. F. A. Fahrenwald (Re-issue 16,556, 22.2.27, of U.S.P. 1,346,188, 25.8.19).—See B., 1920, 601 a.

Apparatus for case hardening. F. J. Commin and S. W. G. Snook (E.P. 265,681, 10.11.25).

Ore concentration (U.S.P. 1,605,172).—See II.

XI.—ELECTROTECHNICS.

Rapid coking of carbon electrodes. G. Schuchardt (Chem.-Ztg., 1927, **51**, 103—104).—In the manufacture of electrodes the use of pitch instead of tar as binding material produces a harder coke, thus increasing the durability of the electrode, since the coke yield from pitch is 40—60%, whereas with tar it is 33%. More rapid coking is also possible, the fluid hydrocarbons of tar making the slow process necessary. In slow coking gradual distillation of the hydrocarbons of high b.p. takes place, so that the coking potentiality is lowered, and the system may be choked with naphthalene and tarry products. In the rapid coking process the electrode, direct from the press, is placed in an incandescent muffle furnace, and the outside shell is coked very quickly, preventing distillation from the interior. The reactions are similar to those in a gas retort, and the gases are used for the direct heating of the muffle. The process takes 3—6 hrs., whereas slow coking occupies 2—3 weeks. It is simpler and cleaner, and more economical in initial cost of plant and in upkeep. W. G. Carey.

Products formed during the working of lead accumulators. II. L. Mazza (Atti R. Accad. Lincei, 1927, [vi], **5**, 117—119. Cf. B., 1926, 953).—*X*-Ray photographs show that in a normally charged or overcharged lead accumulator the paste of the positive plates consists entirely, or almost so, of lead peroxide, whereas when the cell is partly or completely discharged the paste contains a mixture of peroxide and sulphate. T. H. Pope.

Patents.

Electric furnace. Siemens-Schuckertwerke G.m.b.H., Assees. of Heraeus-Vacuumschmelze A.-G., and W. Rohn (E.P. 244,426, 28.10.25. Conv., 15.12.24).—In an electric furnace having heating conductors, *e.g.*, of iron, in the form of metal bars attached to the inner surface of the furnace so that heat is radiated directly into the furnace chamber, the total heat-radiating surface is made equal to or greater than the plane superficial area of the inner surface of the chamber. The metal casing enclosing the furnace may be filled with a neutral, hardening, or cementing gas. J. S. G. Thomas.

Heat-treating [electric] furnace. A. D. Keene, Assr. to Westinghouse Electric & Manuf. Co. (U.S.P. 1,617,396, 15.2.27. Appl., 15.10.23).—A number of spaced and laterally extending openings are arranged in a block of refractory, electrically-conducting material surrounded by a refractory, high temperature-resisting casing. The intermediate opening forms the heating chamber, and a rod extends into each outer opening and co-operates with the walls thereof to produce an arc discharge. J. S. G. Thomas.

Electrodes for electric furnaces. Norske Aktieselskab for Elektrokemisk Industri (E.P. 258,560, 3.8.26. Conv., 19.9.25).—The mantle of a self-baking electrode is made of an easily fusible metal, *e.g.*, aluminium, whilst the ribs are made of a metal, *e.g.*, iron, which is fusible with difficulty, and have a sufficient cross-section to ensure that the voltage drop in them is small. Mantle and ribs are mechanically independent, the ribs being displaceable relatively to the casing and to the electrode in a direction parallel to the axis of the electrode, so that the metal of the ribs does not enter and contaminate the charge. J. S. G. Thomas.

Producing high permeability in magnetic material. Western Electric Co., Ltd. From Bell Telephone Laboratories, Inc. (E.P. 265,642, 8.8.25).—Loading material in strand form containing iron and nickel is heated to above 800°, cooled quickly from at least 600°, mechanically worked to give it the requisite hardness, reheated to about 600°, and then cooled so that its temperature falls about 15°/sec. J. S. G. Thomas.

Processes for forming moulded [insulation] products. Igranic Electric Co. From Cutler-Hammer Manuf. Co. (E.P. 264,978, 4.12.25).—Disintegrated material, *e.g.*, magnesium oxide or alundum, is placed within a non-expanding mould, and further disintegrated by hydration, so that it expands against the walls of the mould and of any object to be encased placed within the mould. The material is finally dehydrated. The process is applicable to the manufacture of casings for electrical resistors. J. S. G. Thomas.

Electrical lighting tube with a filling of carbon dioxide. Patent-Treuhand Ges. für Elektrische Glühlampen m.b.H., Assees. of H. Ewest and W. Schallreuter (G.P. 434,417, 5.2.25).—Carbon dioxide is evolved from magnesium carbonate, beryllium carbonate, calcium carbonate, dolomite, etc., or from mixtures or compounds of magnesium carbonate with other carbonates, placed in the tube, preferably upon the electrodes. The device is used in conjunction with electric lamps of the "daylight" type. J. S. G. Thomas.

Electrical resistor containing silicon and carbon. Gebr. Siemens & Co. (Swiss P. 116,016, 27.2.25. Conv., 12.5.24).—The contact points of resistances containing silicon and carbon are protected by a sprayed coating of chromium–nickel alloy. If desired, the electrodes may be given an undercoat of silver and the chromium–nickel coating covered with silver or aluminium. J. S. G. Thomas.

Gas-filled incandescence lamp. Westinghouse Lamp Co. (Swiss P. 114,997, 20.11.24).—Gas-filled lamps containing oxygen-free organic compounds, *e.g.*, diphenyl, carbazole, anthracene, or organic compounds containing combined oxygen, *e.g.*, Crystal Violet, as purifying media are filled with up to 10% of hydrogen, in order to prevent the formation of carbides upon the filament and to reduce the fragility of the same. The filling gas is freed from moisture, oxygen, and carbon

dioxide by treatment with phosphorus pentoxide, heated copper, and sodium hydroxide.
J. S. G. Thomas.

Manufacture of filaments for electric incandescence lamps. General Electric Co., Assees. of Patent-Treuhand Ges. für Elektrische Glühlampen m.b.H. (E.P. 265,113, 19.11.26. Conv., 10.7.26).—Wire, ribbon, or a spiral filament, *e.g.*, of tungsten, is wound on a former, clamped with the former in a surrounding mould, and then heated at a temperature, *e.g.*, 1300°, sufficient to eliminate internal stresses.
J. S. G. Thomas.

Electron discharge apparatus. British Thomson-Houston Co. Ltd., Assees. of J. C. Warner (E.P. 254,731, 1.7.26. Conv., 1.7.25).—In electron discharge apparatus of the three-electrode type the efficiency of operation is increased by arranging an extra grid between the space-charge grid and the cathode, whereby the resistance between the two latter may be greatly increased by impressing upon the extra grid a voltage lower than that corresponding to the part of the field at which it is located. J. S. G. Thomas.

Production of oxidised cathodes. Süddeutsche Telefon-Apparate-, Kabel-, & Drahtwerke A.-G. (G.P. 434,009, 19.4.21).—A carrier filament coated with metal is treated with a quantity of oxygen sufficient only for the oxidation of the metal layer. L. A. Coles.

Manufacture of hollow conductors. A. L. Mond. From Metallbank u. Metallurgische Ges. A.-G. (E.P. 265,849, 30.6.26).

Removing boiler scale (E.P. 264,551).—See I.

Depositing boron (E.P. 264,953)—See VII.

Moulded compositions (U.S.P. 1,619,692).—See XIII.

Moulded articles (U.S.P. 1,619,758).—See XIII.

XII.—FATS; OILS; WAXES.

Determination of fatty matter in vegetable products. F. Terroine, G. Lepage, J. Véchot, and A. Wolff (Ann. Falsif., 1927, **20**, 79—87).—Prolonged extraction of fatty vegetable material by ether or light petroleum yielded a product which, in the case of material high in fat, approached the sum of the fatty matter and unsaponifiable matter as obtained by Kumagawa's method (B., 1908, 455), but differed from it considerably (15—27%) for material low in fat, owing to some of the fatty acids and particularly the unsaponifiable matter not being extracted by these solvents. D. G. Hewer.

Surface properties of soap solutions. Structure of the surface layer. J. F. Carrière (Chem. Umschau, 1927, **34**, 57—67).—See B., 1926, 332.

Autoxidation of oils. Moureu, Dufraisse, and Chaux.—See II.

Rancidity in lard. Husa and Husa.—See XX.

Patents.

Splitting of natural oils and fats. Allgem. Ges. für Chem. Industrie m.b.H. (G.P. 434,794, 14.3.24).—A separation into unsaturated and saturated or nearly saturated glycerides is effected by means of sulphur dioxide and organic solvents. S. S. Woolf.

Deodorising oils and fats. S. Robertson (G.P. 415,796, 30.5.23).—Fish oils etc. are treated with boiling water in the presence of ammonia or organic bases and turpentine or benzene, its homologues, or chloro-derivatives, until an odourless distillate is obtained.
S. S. Woolf.

Apparatus for refining vegetable and animal oils and fats. E. Herrndorf (G.P. 434,568, 23.9.24).—The vessel in which de-acidification, washing, and bleaching take place is fitted with adjustable fine nozzles, so that distillation with superheated steam and subsequent cooling by a current of air can be carried out in the original vessel. S. S. Woolf.

Still for distillation of fatty acids. Continentale A.-G. für Chemie (G.P. 434,219, 4.2.26).—A still of enamelled acid-resisting metal, fitted with dry steam feed, pitch outlet, and a horizontal disc for preventing frothing, is described. S. S. Woolf.

Apparatus for drying soaps. C. E. Rost & Co. (G.P. 409,129, 23.3.24).—Soap is fed through two rollers, one of which is heated to a greater extent than the other, the large surfaces thus exposed leading to rapid evaporation of water from the soap. S. S. Woolf.

[Perfumed] soaps. E. Weidner (G.P. 434,796, 9.3.24).—The permanence of perfumed soap during storage is improved if the perfuming agents are incorporated with fatty or waxy filling materials and introduced into channels or holes in the soap.
S. S. Woolf.

Enzymatic cleansing materials, toilet articles, etc. H. Fischer (E.P. 265,024, 12.3.26).—Tryptic enzymes, or mixtures of enzymes in which the tryptic component naturally preponderates, are dehydrated, preferably *in vacuo*, at 45°, to a water content of not more than 10%, before incorporation with soaps or other cleansing agents (also suitably dehydrated), water softeners, and substances intended to reduce surface tension. By restricting the amount of water present enzyme activity may be retained for prolonged periods. S. S. Woolf.

Removal of free fatty acids from substances containing fats. Soc. Anon. des Établissements Rocca, Tassy, & de Roux (F.P. 609,842, 5.5.25).—The greater part of the free fatty acids present in a fatty substance is removed by a preliminary extraction with a mixture of acetone and water before the usual extraction and pressing. S. S. Woolf.

Separation of solid resinous substances and oleaginous fatty acids from tall oil. Chem. Fabr. Dr. H. Noerdlinger A.-G. (G.P. 434,924, 15.3.24).—Solutions of tall oil in mineral oils or other solvents unaffected by concentrated sulphuric acid are warmed with the requisite amount of that acid to cause resin to separate. Oleaginous fatty acids are recovered from the solution. S. S. Woolf.

Acetylenation of fatty and other substances. L. Bourgoin (E.P. 265,677, 9.11.25).—See U.S.P. 1,567,785; B., 1926, 288.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Use of vanadium compounds as driers. R. Swehten (Farben-Ztg., 1927, 32, 1138—1139).—The drying times and behaviour on drying of various mixtures of raw linseed oil and driers were observed. The fused resinates and linoleates of vanadium, lead, manganese, and cobalt were used in concentrations giving metal contents of 0·1, 0·2, and 0·3%, respectively, in the mixtures. The average drying powers of the four metals under these conditions are, roughly, as 5 : 1 : 2 : 7 in the order named. Despite its relatively powerful drying action, vanadium does not give rise to the surface drying, film wrinkling, and brittleness associated with the use of cobalt or manganese driers, but has the general good qualities of lead as a drier, with enhanced drying power. Its use, however, is restricted to dark-coloured products owing to the "browning" of films induced by it. S. S. Woolf.

Specific gravity, bulk density, and opacity of lithopone. W. N. Hirschel (Chem.-Ztg., 1927, 51, 140—141).—Samples of lithopone containing over 30% of zinc sulphide were prepared and mixed with barytes to reduce the zinc sulphide content to 30% in each case. The sp. gr., bulk density, and opacity (reduction with ultramarine blue, 20 : 1) were compared with those of normal lithopones, barytes, and white lead, but no relationship between the opacity and the remaining constants could be established. In general, the admixed lithopones were not as strong as the precipitated samples, the opacity of barytes being found to be one-fifth of that of the standard lithopone. S. S. Woolf.

Detection of resins according to Brauer's method. E. Fonrobert and K. Pistor (Chem.-Ztg., 1927, 51, 139—140).—Brauer's method for distinguishing between various resins by means of ammonium phosphomolybdate etc. (cf. B., 1926, 595) is examined. The tabulated results obtained by the authors are often in disagreement with those of Brauer, and the method is considered to be insufficiently selective. For the recognition of rosin (colophony) the Brauer reactions show no advantages over the Storch–Morawski (Liebermann) reaction. S. S. Woolf.

Lake dyestuffs. Harrison.—See IV.

Resin from kauri wood.—See V.

Patents.

Paints for fishing nets. S. Tomioka (U.S.P. 1,617,426, 15.2.27. Appl., 24.3.26).—A mixture of equal parts of linseed and sardine oils with 0·5% of lead oxide and 0·6% of manganese resinate is heated at 260° for more than 10 hrs. Copal is melted and 4% of lime and 10% of sulphur are added. 40 pts. of the resin component and 60 pts. of the oil component are finally heated together. S. S. Woolf.

Manufacture of lead compounds. Commonwealth White Lead and Paints Proprietary Ltd. (E.P. 249,809, 29.9.25. Conv., 30.3.25).—Metallic lead is reduced to a fine dust by an air blast to which it is continually exposed by the action of the rotary furnace in which it is being melted. The particles of lead dust are precipitated from the air current and mixed with water to form a coherent, plastic mass, which is then moulded into briquettes. After optional ageing, the lead is converted into such compounds as red lead, white lead, lead sulphate, etc. (the design of the briquettes rendering all metal particles accessible to reagents). S. S. Woolf.

Zinc sulphide pigment. L. Mellersh-Jackson. From New Jersey Zinc Co. (E.P. 250,581, 6.4.26).—A mixture of zinc oxide (average particle size not exceeding 0·25μ; cf. E.P. 219,423; B., 1924, 787) with excess of sulphur is heated at 600—800° and the resulting product cooled under non-oxidising conditions. A second heating at the reaction temperature improves the colour of the zinc sulphide so obtained, which is practically pure, and is a satisfactory white pigment of average particle size 0·5μ. S. S. Woolf.

Sizing composition for coating purposes. F. R., M., and A. Hervé (U.S.P. 1,615,584, 25.1.27. Appl., 16.7.25. Conv., 16.6.25).—Casein, or similar albuminoid substance, is dissolved in a slight excess of sodium bicarbonate solution, a small quantity of powdered aluminium, or other powdered metal, or alloy, is added, and the product is employed as a sizing composition for surfaces, *e.g.*, glass, wood, to improve the durability of paints and varnishes. T. S. Wheeler.

Manufacture of condensation products from phenols and formaldehyde. E. J. P. C. de Jarny (E.P. 265,033, 30.3.26).—If the amount of alkaline earth used as catalyst in phenol–formaldehyde condensations be increased to more than 15% of the formaldehyde solution used, the plastic resinous masses obtained remain malleable in the cold at all stages of the subsequent "curing" short of the final one. Solubility in acetone and alcohol, however, decreases as the reaction proceeds. S. S. Woolf.

Purification of resinous phenol–aldehyde condensation products. Bakelite Ges.m.b.H., Assees. of F. Seebach (G.P. 432,727, 7.3.25. Addn. to 431,514; B., 1926, 889).—Crude phenol–aldehyde condensation products, containing 5—15% of free phenol, are purified by agitation with water and oxides or hydroxides of the alkaline earths, magnesium, aluminium, etc., or substances producing them, and subsequent washing. Typical materials used are: milk of lime, magnesia, sodium aluminate, calcium zincate, zinc chloride–ammonia, ammoniacal copper sulphate, the molecular compound of lime and glycerin, calcium saccharate, calcium sulphide, etc. The production of fine suspensions not readily separated by centrifuging etc. is remedied by the flocculating action of aluminium α-naphthylamine-4-sulphonate. Dry processes of a similar nature are also described. S. S. Woolf.

Preparation of resinous condensation products. British Thomson-Houston Co., Ltd., Assees. of E. S. Dawson, jun. (E.P. 252,394, 21.5.26. Conv., 23.5.25).—In condensations of the "glyptal" (glycerol–phthalic anhydride) type, a relatively small proportion of sulphuric acid is used, sufficient, however, to modify the normal chemical reaction (*i.e.*, sulphuric acid being absent). Fatty acids, preferably derived from linseed or tung oils, are next added, and heating is continued until a homogeneous resinous condensation product results. The

latter may be used in solution in high-boiling solvents, *e.g.*, glycol diacetate or ethyl phthalate, plasticisers such as castor or tung oils being incorporated.

S. S. WOOLF.

Manufacture of vitreous masses. E. HOPE (E.P. 264,550, 26.10.25).—Dialkyl esters of itaconic acid are polymerised by heating in the absence of basic substances at 60° or higher, alone or mixed with fillers and/or colouring matter. The anti-catalytic action of bases is utilised for the stabilisation of the esters during storage (cf. E.P. 254,668; B., 1926, 747). S. S. WOOLF.

Producing acid-proof, adherent, and repairable coatings from artificial resins. C. ten D. KOOLMAN (E.P. 253,531, 8.6.26. Conv., 9.6.25).—An artificial resin film is built up of alternate layers of the resin itself and of the resin admixed with heat- and acid-resistant materials, *e.g.*, kieselguhr, asbestos, graphite, etc. (layers being applied while the previous coating is still able to swell and bind), and the whole mass is "cured." Any damage to the film can then be repaired by application of fresh artificial resin, which will adhere to the rough intermediate layers suitably exposed by grinding at an angle, whereas it would not do so to the resin itself. S. S. WOOLF.

Making moulded compositions. L. E. BARRINGER and C. F. PETERSON, Assrs. to GENERAL ELECTRIC Co. (U.S.P. 1,619,692, 1.3.27. Appl., 10.4.25. Renewed 26.7.26).—See E.P. 203,232; B., 1923, 1031 A.

Process of making moulded articles. C. F. PETERSON, Assr. to GENERAL ELECTRIC Co. (U.S.P. 1,619,758, 1.3.27. Appl., 14.4.25).—See E.P. 250,942; B., 1927, 119.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Production of mixtures of rubber with inorganic and organic vulcanisation accelerators. O. FISCHER (Austr. P. 103,903, 17.12.24).—Homogeneous mixtures of rubber, sulphur, and a vulcanisation accelerator are prepared by mixing the sulphur and the accelerator with separate portions of rubber and then rapidly mixing the two mixtures. L. A. COLES.

Combining cellulose and rubber (U.S.P. 1,617,495).—See V.

XV.—LEATHER; GLUE.

Proposed method for the determination of the enzyme value of artificial bating materials. V. KUBELKA and J. WAGNER (J. Soc. Leather Trades' Chem., 1926, **10**, 432—443).—The bating material is taken up with distilled water at 37°, filtered, the insoluble residue dried and weighed. It is examined microscopically for sawdust or ground straw and chemically after ignition for weighting materials. Ammonium sulphate and chloride should be determined in the filtrate. The enzymic activity of the latter should be determined on a 0·05% casein solution in a series of 10 tubes arranged in a thermostat maintained at 40°. Ten 1 c.c. graduated pipettes fixed in a frame are connected with each other at their upper ends by a T-tube, each being fitted with a stopcock. An automatic pipette holding 1000 c.c. in its bulb portion and 5 c.c. in the stem is used to deliver 5 c.c. of the casein solution to each of the ten tubes, 1 c.c. of distilled water being added to each tube from a series of ten pipettes. When the tubes and contents have reached 40°, measured quantities 0·1—1·0 c.c. of enzyme solution are run in from a second series of pipettes. The tubes are kept at 40° for 30 min. after running in the enzyme solution, cooled to 15—20°, and 0·5 c.c. of 0·25% acetic acid solution is run into each tube from a third series of pipettes. The tube which shows only a very faint opalescence and no Tyndall effect with transmitted light marks the end-point. The amount of casein digested by 1 kg. of the bate can then be calculated. This apparatus enables the comparative digestions to commence simultaneously, and thus avoids errors due to separate pipetting.

D. WOODROFFE.

Extraction of tanning materials using a modified Teas' extractor. H. E. WILLIAMS (J. Amer. Leather Chem. Assoc., 1927, **22**, 97—102).—A 2500 c.c. flask is fitted with a Teas' extractor (cf. B., 1905, 1055) the cock of which is soldered tight, and in the side of the tube above the cock a smaller tube is soldered to allow a thermometer to be inserted into the material to be extracted. A flanged copper pipe is soldered to the top of the extractor, and into this pipe is set a stopper carrying a condenser, the upper end of which is connected to a glass tube set in a 3-hole stopper in the neck of a 2-litre receiver attached to the delivery tube of the extractor through a tube set in the second hole in the stopper. The third hole is fitted with a tube leading to a vacuum break, which is connected by means of a T-tube to a filter pump. To extract at temperatures below 100°, the pump is started and the vacuum break adjusted so that the temperature of the boiling water is that desired. The vacuum break can also be used as a pressure gauge for extracting under pressure at temperatures below 100°. D. WOODROFFE.

Leather dyeing. V. H. SALT (J. Soc. Leather Trades' Chem., 1926, **10**, 468—470).—Samples of skivers were tanned with 12 different vegetable tanning materials and one synthetic tannin respectively, and then dyed with 0·25% and 1% titanium potassium oxalate, 0·5% ferrous sulphate, a mixture of 5% ammonium acetate and 0·5% ferrous sulphate, and various acid and basic dyestuffs respectively. The treated leathers showed little variation in shade between one tannage and another, except that due to the original colour of the leather. Valonia-tanned leather took up less dyestuff. Only sumach-tanned leather gave a grey with iron; other tannages showed no tendency to a black.

D. WOODROFFE.

Effect of time and tannin on the analytical results, yield, cut, and colour of [sole] leather. CASTE and E. PARSY (J. Soc. Leather Trades' Chem., 1926, **10**, 471—476).—Samples of sole leather which had been tanned for 15 days in liquors (*d* 1·004—1·045) consisting of $\frac{2}{3}$ quebracho and $\frac{1}{3}$ chestnut extracts, then for 36 hrs. in equal parts of quebracho and chestnut extract liquors (*d* 1·075) were dried, and submitted in sets of three to immersion in chestnut extract (*d* 1·074) at 17—20° (A) alone, (B) with 0·8% of added sodium bisulphite solu-

tion (d 1·320), (C) with ground oak bark, and (D) with a solution of 10% acetic acid respectively. Samples were withdrawn in each case after 11, 30, and 60 days' immersion and analysed. The increase in the weight of the leather was less with ground bark than without it. The acetic acid treatment made no difference. The degree of tannage was greatest with the bark and extract. The colour with bisulphite and chestnut extract was better than that of chestnut alone or with bark. The cut was slightly darker with the oak bark. The analytical results show that the use of sodium bisulphite with chestnut extract as a vatting liquor is superior to chestnut extract alone or with oak bark as regards yield of leather, the preservation of the liquors, and the colour of the tannage. D. WOODROFFE.

Acidity of chrome[-tanned] leather. K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1927, **22**, 60—69).—Treatment of moist or finished chrome-tanned leathers with a 3—5% pyridine solution for 16 hrs. removes the sulphuric acid combined with the collagen, but does not affect the sulphato-chromium complex. The two-bath chrome-tanned leather contained a complex of higher acidity than that of a one-bath leather tanned in a moderately acid chromium sulphate at relatively low concentration and without any addition of sodium sulphate. The addition of the latter to a one-bath chrome-tanning liquor, followed by heating the same, produced a chromium–collagen complex of similar or higher acidity than that of the two-bath chrome-tanned leather. It is suggested that the electronegative chromium complexes associated with the basic groups are more easily hydrolysed than the ordinary sulphato-chromium–collagen complexes. Solutions of chromium chloride of moderate concentrations and 50—70% acidity gave leathers containing a chlorochromic complex of about 10% acidity. With extremely basic chlorides the acidity was only 4·2%. Addition of neutral chlorides to the basic chromium chloride tanning liquors did not cause much increase in the acidity of the chloro-complex. Chromium sulphate liquors gave a diminished acidity of the sulphato-chromium–collagen complex with increased basicity. Addition of sodium sulphate to the sulphate liquors increased the acidity of the complex, *i.e.*, caused a migration of sulphate groups into the internal sphere. A basic chromium sulphate liquor (acidity 60%) containing organic acids from the reduction process gave a leather with acidity 62·1% before and 19% after pyridine treatment. The organic acids must have replaced some of the sulphate groups in the complex, since the latter is usually 30%. The addition of formates and acetates to chrome-tanning liquors diminished the acidity of the complex from 29·4% to 16·0% in a 63% acid chromium sulphate. Acetates diminish the tanning power of both chloride and sulphates of chromium. Formates added to sulphate liquors to give p_H 5·0 yielded an excellent tanning agent, but with decreased chromium fixation; in chloride liquors in quantities up to 1—1·5M there was nearly double the fixation of chromium. These experiments prove the existence of acidic groups in direct combination with chromium in chrome-tanned leathers. D. WOODROFFE.

New method of chrome tannage. M. SHIMIDZU (J. Amer. Leather Chem. Assoc., 1927, **22**, 93—97).—The pickled hides are drummed with 3% of potassium dichromate and 150% of water, hung up to drain, transferred to a fuming chamber of sulphur dioxide, left until completely reduced, and afterwards neutralised in the usual way. The leather produced compares favourably with that tanned by the ordinary methods. There is an economy in time, labour, and material. Only 1% of sulphur (on the hide weight) is required to be burnt to produce the sulphur dioxide necessary, instead of 3% of acid and 6% of sodium thiosulphate. D. WOODROFFE.

"Ageing" of chrome[-tanned] leather. K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1927, **22**, 102—105).—Samples of leather tanned with a one-bath chrome liquor of the cationic type and by the two-bath tannage using sodium bisulphite as the reducing agent, respectively, were allowed to age, and portions analysed after different periods to determine the acidity of the sulphato-chromium–collagen complex in the leather. The p_H value of the water was determined after it had been 24 hrs. in contact with the aged leathers. The chromium complex in one-bath chrome leather had 29·5% acidity without ageing, and diminished to 24·4% after 2 weeks' ageing. The p_H value of the water was 3·12 on the original sample, and 2·96 after 6 weeks' ageing. The acidity of the chromium complex in two-bath chrome leather was 27·8%, and remained practically constant during ageing. The p_H value of water in contact with the samples of two-bath chrome-tanned leather diminished from 3·98 in the original, to 3·0 after 3 weeks' ageing. The acidity of the sulphato-chromium–collagen complex in two-bath chrome leather was only slightly greater than that of the one-bath leather. The amount of acid removed by pyridine treatment was greater from two-bath than from one-bath tanned leathers, but there is 7 times as much hydrolysis with the latter. The acidity of the cationic chromium complex was about the same for both tannages. Probably the sulphato-groups in the anionic complex are more labile, and those in the cationic complex may serve as connecting links with the protein. On ageing, chrome-tanned leather does become more acid. D. WOODROFFE.

PATENTS.

Tanning agents. I. G. FARBENIND. A.-G. (G.P. 433,163, 26.8.24. Addn. to 420,647; B., 1926, 456).—The oxidation products obtained by treating fossilised vegetable matter with oxidising agents such as nitric acid or nitrous fumes are replaced by the oxidation products of lignite, brown coal, peat, etc. The formaldehyde condensation products of naphthalenesulphonic acids or their salts are wholly or partially replaced by other synthetic tannins containing one or several sulpho-groups in the molecule. The dry oxidation products obtained by the action of nitric acid on lignite are mixed with the sodium salt of the formaldehyde condensation product of naphthalenesulphonic acid. This mixture gives a bright reddish-brown leather with a good feel. By replacing the sulphonic acid partially or entirely with the condensation products of naphthalenesulphonic acid and sulphur, or sulphur and phenol, or glycollic acid,

or cresolsulphonic acid and formaldehyde, and neutralising to a suitable acidity, the product tans a bright coloured leather. A mixture of equal parts of a 20% solution of the tannin obtained by oxidising brown coal with nitric acid, and a concentrated solution of the formaldehyde condensation product of cresolsulphonic acid, after diluting with water, gives a well-filled, pale brown coloured leather. D. WOODROFFE.

Tanning agents. I. G. FARBENIND. A.-G. (G.P. 433,162, 29.6.24).—Fossilised material of vegetable origin, *e.g.*, coal, brown coal, humus coal, or peat, is treated to remove the iron content by stirring the finely-powdered material with 20% sulphuric acid and heating. The product is filtered, washed with water, and the damp or dried residue used in the manufacture of tannins. The removal of the iron increases the yield of tannin when the material is oxidised, and the product gives brighter coloured leather, especially when mixed with vegetable tannin extracts. D. WOODROFFE.

XVI.—AGRICULTURE.

Determination of the manurial requirement of soils. GERLACH (Landw. Jahrb., 1926, 339—368; Bied. Zentr., 1927, **56**, 58—60).—Comparison is made of the methods of Lemmermann, Neubauer, and Mitscherlich. All soils, except peats, possessed insufficient reserves of active nitrogen compounds, and in most cases the amounts of available potash and phosphate present were such that ordinary dressings of farmyard manure sufficed. Peat soils were usually deficient in potash. Light sandy soils needed lime, especially for barley and wheat and on pastures, and were usually lacking in phosphate. Farmyard manure was not adequate to remedy this deficiency for wheat crops. Interrelationships between the total phosphate, citric-soluble phosphate, phosphate utilised by crops in field trials, and the root-soluble phosphate (Neubauer) were not apparent. The value of adequate basal manuring with phosphate and potassium salts and the application of nitrogenous material as a top-dressing is emphasised.

A. G. POLLARD.

Nitrogen-manuring, and the profitable cultivation of cereals. O. NOLTE and R. LEONHARDS (Mitt. deut. Landw. Ges., 1926, **27**, 563; Bied. Zentr., 1927, **56**, 60—63).—Field trials with rye showed profitable results from the use of ammonium sulphate. Best results followed early application of the fertiliser. When used as a spring dressing the optimum quantity of fertiliser remained the same, but the yields were smaller. When applied in early summer, ammonium sulphate produced still smaller yields, and no relationship was here apparent between the quantity of fertiliser used and the crop return. A. G. POLLARD.

Phosphate manuring. O. NOLTE and R. LEONHARDS (Mitt. deut. Landw. Ges., 1926, **34**, 701; Bied. Zentr., 1927, **56**, 63—66).—Records of field trials of various phosphate fertilisers with different crops are recorded. For winter-sown cereals better crops resulted from spring application of phosphates than when applied just previous to sowing. A. G. POLLARD.

Determination of mineral nitrogen in fertilisers. C. H. JONES (Ind. Eng. Chem., 1927, **19**, 269—271).—The following method gives reliable results in the determination of nitrate nitrogen in presence of cyanamide or carbamide. 4 g. of fertiliser are dissolved in 200 c.c. of water and filtered. In one 50 c.c. portion ammoniacal nitrogen is determined by distillation with magnesium oxide. Another 50 c.c. are warmed with 2 g. of reduced iron and 10 c.c. of dilute sulphuric acid, diluted, cooled, and 100 c.c. of concentrated sodium hydroxide solution added. It is then at once distilled and the ammonia determined. A correction blank is carried through without any reduced iron. The difference accurately represents the nitrogen present as nitrate. The large excess of sodium hydroxide is necessary to minimise any action of the hydrogen other than on the nitrate. Tests on mixtures prepared in the laboratory gave much more accurate results than the U.S. official procedure. C. IRWIN.

Oxidation of organic matter and nitrification in sterilised soils kept for a long period in contact with oxygen. E. PARISI and G. CARBONCINI (Annali Chim. Appl., 1927, **17**, 45—52. Cf. B., 1926, 641).—Degradation of organic matter and nitrification are favoured by the presence in the confined atmosphere of a considerable proportion of oxygen. Plant life being hampered or impossible under such conditions, the experiments now described aimed at ascertaining if the oxidation phenomena observed are due to the microflora of the soil or to chemical action of the concentrated oxygen. The oxidation is due principally to biological phenomena, which are always accompanied by chemical phenomena. Soil repeatedly sterilised in an autoclave at 130° absorbs far less oxygen than that sterilised by treatment with antiseptics. When the space containing the soil is limited, the atmosphere is free from carbon dioxide at the end of the experiment. Hence, at the ordinary temperature and in presence of antiseptics, the chemical oxidation of the organic residues of the soil is caused mainly by substances which are altered when heated, and are, perhaps, analogous to those occurring in pulped leaves (cf. Ciamician and Ravenna, A., 1919, i, 58, 140). The formation of nitrates is arrested completely, or almost so, by the action of antiseptics, so that the oxidation of ammonia in soil in contact with oxygen is due to micro-organisms. T. H. POPE.

Properties of soil colloids. A. N. SOKOVOLSKII (Pochvovedenie (Russian), 1924, **19**, 59—79).—Elimination of calcium from soils by replacement brings about a condition whereby extraction of such soils with distilled water brings into pseudo-solution some of the soil colloids. The structure of the soil is destroyed by such treatment. By continuous extractions and decantations a certain fraction of peptised mineral and organic substances may be separated. By treating the residue with hydrogen peroxide another peptised fraction may be obtained. The first fraction is known as the active and the second as the passive. The absorbed calcium serves as a coagulator of the sols present, and liming serves the purpose of preserving the soil colloids.

CHEMICAL ABSTRACTS.

Colloid chemistry of soils. E. TRUOG (Third Colloid Symposium Monograph, 1925, 228—240).—A new ultrafilter and a colorimetric method of determining p_H in soils are described. CHEMICAL ABSTRACTS.

Power of soils to absorb water from air. F. J.

Alway (Third Colloid Symposium Monograph, 1925, 241—246).—Davy's view (1814) that the power of soils to adsorb water from air is much connected with fertility seems to be fully substantiated, but the ability of soils to adsorb water vapour is not a trustworthy measure of their colloid content. Chemical Abstracts.

Adsorption by activated sugar charcoal, with particular reference to soil acidity. E. J. Miller (Mich. Agric. Exp. Sta. Tech. Bull., 1925, **73**, 1—60).—A discussion of the nature and origin of soil acidity, based on work previously published (A., 1922, ii, 741; 1923, ii, 464; 1924, ii, 734; 1925, ii, 656; Mich. Agric. Exp. Sta. Quart. Bull., 1925, **8**, No. 1, 37). The charcoal and soil systems are similar, but not perfectly analogous, in behaviour. Hydrolytic adsorption of the acids of salts with loss of the bases by leaching, promotion of "negative" adsorption of bases by surface-tension effects, promotion by carbon dioxide of the adsorption of acids, displacement of adsorbed acids by neutral salts, and the irreversibility of the adsorption process with resulting apparent insolubility of such adsorbed acids, have been demonstrated for charcoal.

Chemical Abstracts.

Significance of nitrogen in soil organic matter relationships. F. J. Sievers and H. F. Holtz (Wash. Agric. Exp. Sta. Bull., 1926, **206**, 1—43).—Nitrogen accumulates in a soil only as a result of legume fixation, free fixation, and precipitation. The nitrogen : carbon ratio is stable, and the contents of these two elements indicate that of the organic matter. Abundant precipitation and high temperature, which promote the accumulation of organic matter, are also most effective in promoting its decomposition. In the process of decomposition of plant residues in the soil there is a tendency for the nitrogen : carbon ratio to be narrowed until it approaches that of the micro-organisms responsible for the decomposition. For the measurement of the decomposition the carbon dioxide evolved and the nitrate accumulated were measured.

Chemical Abstracts.

Mobility of soil compounds, and influence of calcium thereon. K. K. Gedroiz (Nosovsk [Russia] Agric. Exp. Sta. Bull., 1926, **43**, 1—18).—The calcium ion is beneficial in both acid and alkaline soils, in the former by preventing the hydrogen ions from rendering the soil unsaturated, and in the latter by preventing the sodium from getting into the colloidal fraction of the soil. Chemical Abstracts.

Improved stain for bacteria in soil. H. J. Conn (Stain Technol., 1926, **1**, 125—128).—A suspension of soil in a 0·015% gelatin solution is dried on a slide, treated for 1—3 min. in 40% acetic acid, washed, and dried on a boiling water-bath, and while still on the bath is stained for 1 min. with 1% aqueous rose bengal.

H. W. Dudley.

Mechanical analysis of soil by the method of decantation with water. M. Filatov (Pochvovedenie [Russia], 1925, **20**, 33—41). Chemical Abstracts.

Patents.

Producing artificial [nitrogenous] manures. Siemens & Halske A.-G. (E.P. 245,768, 5.1.26. Conv., 9.1.25.).—Carbon monoxide, ammonia, and chlorine are allowed to react with each other in such a manner as to produce nitrogenous compounds. In the preferred method phosgene is first produced from carbon monoxide and chlorine, and then allowed to react with ammonia or an ammoniacal solution. By varying the proportions of the reacting materials a mixture of carbamide and ammonium chloride, or a mixture of ammonium chloride, carbamide, and such compounds as cyanamide, cyanuric acid, etc. may be obtained. G. W. Robinson.

Manufacture of alkali-nitrogen fertilisers having carbamide as their base. J. Breslauer, Assr. to Comp. de l'Azote et des Fertilisants S.A. (U.S.P. 1,618,266, 22.2.27. Appl., 22.6.25. Conv., 26.6.24).—See F.P. 600,016; B., 1926, 417.

Manufacture of nitrogenous fertilisers. E. H. Richards and H. B. Hutchinson, Assrs. to Adco Ltd. (U.S.P. 1,619,679, 1.3.27. Appl., 15.4.24. Conv., 23.4.23).—See E.P. 219,384; B., 1924, 801.

XVII.—SUGARS; STARCHES; GUMS.

Factors influencing char filtration [of sugar solutions]. E. W. Rice and G. W. Murray, jun. (Ind. Eng. Chem., 1927, **19**, 214—215).—Aqueous solutions of various salts which might be present in raw sugar were percolated through refiners bone char at the rate of 150 c.c./hr., and successive 50 c.c. portions tested by conductivity determinations. The temperature was maintained at 77°. The adsorption varied from over 95% with calcium hydrogen phosphate to practically nil, even in the first portion, with sodium chloride. This was from a 2% solution in each case. The results are disturbed by the adsorption of water and the variation of adsorptive power with different concentrations, but it is held that complete purification of raw sugar by the use of bone char alone is impossible.

C. Irwin.

Nipah palm. Further investigations on tapping and preservation of juice. J. H. Dennett (Malay. Agric. J., 1926, **14**, 375—383. Cf. B., 1923, 620 a).—Figures for the yields of juice per month for 20 months and the effects of tapping on the fruiting of the palms are given. In order to inhibit preliminary natural fermentation in the field, numerous experiments on the preservation of the juice with sulphuric acid have been conducted. Measurements of the rates of decrease in sugar content over a period of days in juices having concentrations of acid varying from 0·25N to 0·0125N, and contained in vessels open to the air, thus allowing access of any fermentation spores, showed that the above concentrations of acid caused inversion of all the sugar content in about 20 hrs., after which fermentation proceeds fairly rapidly in those juices containing weak acid, whilst in the samples containing strong acid the sugar content remains constant over a period of 3 days. The conclusion is reached that, economically, it is preferable to use the stronger acid because the value of the alcohol lost decreases much more rapidly than the value of the sulphuric acid used increases.

E. H. Sharples.

Speed of absorption of moisture by granulated sugar. K. K. Shiiko-Parkhomenko (Zapiski [Russia], 1926, **3**, 291—294; Chem. Abstr., 1927, **21**, 189).—

Absorption of moisture increases with time, and is proportional to the vapour pressure. In general, the fineness of the crystalline state determines the speed of absorption; in part, the speed increases proportionally to the size of the crystals. A. A. ELDRIDGE.

PATENTS.

Macerating sugar-containing materials. N. NOBEL (U.S.P. 1,617,962, 15.2.27. Appl., 18.8.26. Conv., 26.9.24).—The material is conveyed in a mass through a course, and macerating liquid is poured upon it at a number of stages in the course. The liquid is allowed to drip through, the drippings being collected and in part re-poured upon the mass. H. HOLMES.

Production of tricalcium saccharate of low lime and high sugar content from cold molasses or impure sugar solutions. C. STEFFEN, JUN. (Austr. P. 103,918, 13.3.25).—The molasses or sugar solution, immediately after the addition of powdered lime and before introduction into the concentrating vessel, is thoroughly mixed by centrifuging. The amount of lime required is considerably less than in the usual precipitation process. F. R. ENNOS.

Production of rice starch specially suited for the manufacture of glucose. H. KAUTZ, and SOC. ANON. PRODOTTI INDUSTRIALI (Swiss P. 115,305, 1.8.25).—Rice starch, from which dilute sodium hydroxide will remove no more protein, is treated with dilute ammonia solution. The protein content of the starch so treated does not exceed 1%. F. R. ENNOS.

Strainer for sugar juices and the like. S. S. PECK (U.S.P. 1,619,484, 1.3.27. Appl., 27.8.23. Conv., 13.1.23).—See E.P. 208,928; B., 1924, 226.

Beet molasses (F.P. 608,390).—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Relationship between the yield of extract from barley and from malt. E. EHRICH (Allgem. Brau- u. Hopfenztg., **66**, 1385—1388; Chem. Zentr., 1926, II, 2643).—The extract obtained from malt by the Congress method, although lower by 1% than that obtained by the decoction method, exceeded the extract derived from the corresponding barley by amounts varying from 2—3%. The extract from the barley was determined by Pavlovski's method, with an additional digestion for 1 hr. under a steam pressure of 0·4—0·5 atm. A barley which gave 79·3% of extract by the Pavlovski method yielded 81·1% of extract by the extended treatment proposed by the author. C. RANKEN.

Detection of fruit wine in grape wine. A. HEIDUSCHKA and C. PYRIKI (Chem.-Ztg., 1927, **51**, 129).—The reaction of wines with Röttgen's reagent (ammoniacal copper acetate solution; cf., B., 1927, 24) depends on the content and nature of the tannins, which are generally more abundant in fruit wines than in grape wines. In the case of German wines, which cause no colour change in the reagent, adulteration with 15% or more of fermented apple or pear must can be detected, but even with 30% of berry wine the change from blue to green is not very marked. The behaviour of certain Spanish, Italian, and French wines towards the reagent is similar to that of fruit wines. The method affords definite evidence of the absence of cider or perry if the wine remains distinctly blue on treatment with the reagent. F. R. ENNOS.

PATENTS.

Artificial malt product suitable for use in brewing and bread-making. E. A. FULLER (E.P. 264,890, 22.9.25).—To cereal flour, mixed with water to form a liquid of creamy consistency, is added a small proportion of malt and the temperature maintained at 60° for about 3 hrs. After dilution, the liquid is evaporated to dryness on heated rollers, and the malted product scraped off in the form of flakes. F. R. ENNOS.

Process for making lactic acid and lactates. S. E. FAITHFULL (E.P. 265,336, 8.12.25).—See U.S.P. 1,569,221; B., 1926, 252.

Clarification of beverages. J. DUCLAUX (U.S.P. 1,619,016, 1.3.27. Appl., 5.12.22. Conv., 6.12.21).—See E.P. 190,143; B., 1923, 1039 A.

XIX.—FOODS.

Chemistry of New Zealand wheats and flours. II. Hydration capacity of gluten from some local samples. L. D. FOSTER (New Zealand J. Sci. Tech., 1926, 8, 299—304).—Hydration capacities of the glutens of some 32 samples of straight-grade flours of various New Zealand wheats were taken as the number of grams of water taken up by 100 g. of moist gluten, after being in 0·02*N*-hydrochloric acid for 50 min. No close relation was found between hydration capacity and loaf-volume or the quality of the gluten as judged by visual inspection. "Strong" flours had high hydration capacities, whilst those with values below 50% were, with one or two exceptions, amongst the flours considered as "weak." Texture of loaves varied fairly consistently with hydration capacity. D. G. HEWER.

Elimination of sodium and chlorine in cows' milk. L. BARTHE and E. DUFILHO (Ann. Falsif., 1927, **20**, 88—91).—Adulteration of milk by sodium chloride or other salt of sodium may be detected from a consideration of the sodium and chlorine content, since 1 litre of milk from a healthy cow, fed ordinary rations, was never found to contain more than 0·5 g. of sodium or 1·5 g. of chlorine. These figures were not exceeded when the cow was fed 25 and 50 g. of marine salt at successive intervals. D. G. HEWER.

Determination of amount of connective tissue in meat. H. H. MITCHELL, R. L. ZIMMERMAN, and T. S. HAMILTON (J. Biol. Chem., 1927, **71**, 379—387).—The soluble muscle proteins are removed from meat by repeated maceration of the minced material with cold water. The insoluble residue is then heated in the autoclave with water for 2 hrs., and the solution filtered; the nitrogen of this filtrate represents the collagen of the tissue; the precipitate is further subjected to tryptic digestion for 16 hrs. and the nitrogen of the final insoluble residue determined; the latter represents the elastin. Figures obtained by this method are given for various samples of meat. C. R. HARINGTON.

Determination of acid and fat content of casein. J. MARCUSSON and M. PICKARD (Chem.-Ztg., 1927, **51**,

104).—The process of Lunge and Berl for the determination of acid in casein, and also the alkali method of Ulex (B., 1925, 734) are inaccurate, because the amount of shaking and the temperature modify the results, and no account is taken of albuminous substances combined with alkali which are extracted by the water or alkali, whilst the alcohol process of Höpfner and Jaudas (B., 1925, 420) gives low results owing to the retention of lactic acid in the granules of the coagulated casein. For the determination of acid, 5 g. of casein are ground to a paste with 5 c.c. of water, and, after 15 min. to ensure the extraction of the lactic acid from the casein granules, the mixture is ground with sand and extracted with ether in a Soxhlet apparatus. The ethereal solution is shaken with two or three amounts of water, and the lactic acid is titrated in the separated aqueous portion, the fatty acids being titrated in the ethereal solution with standard alcoholic alkali, using phenolphthalein as indicator. The fat is determined in the ethereal solution after the titration with alkali by shaking twice with water to remove the salts formed, distilling off the ether, and weighing the residue. This gives the neutral fat to which is added the fatty acid figure, to obtain the total fat content. Caseins examined gave the following results:—Lactic acid, 1·6—3·9%; fatty acids, 0·8—4·0%; neutral fat, 0·8—4·2%; and ash, 3·1—3·7%.

W. G. Carey.

Determination of common salt in foodstuffs. F. Mach and W. Lepper (Landw. Versuchs-Stat., 1927, **105**, 205—208).—In ashing foodstuffs, chloride may be lost. Determination of chloride in extracts of foodstuffs by customary methods is inaccurate owing to obscured end-points in volumetric work. For accurate results 10 g. of material are shaken with about 400 c.c. of water for 15 min. and treated with 5 c.c. of 10% tannic acid solution and 10 c.c. of a 10% ferrous sulphate solution, previously oxidised with sulphuric acid and hydrogen peroxide. The mixture is made slightly alkaline by the addition of saturated sodium carbonate solution (added till colour changes), 1 c.c. of 3% hydrogen peroxide solution is added, followed by a slight excess of acetic acid. The volume is adjusted to 500 c.c., and after filtration the chloride in 50 or 100 c.c. of the filtrate is determined by Volhard's method, 10 c.c of ether being added to aid the clotting of the precipitate.

A. G. Pollard.

Patents.

Apparatus for testing doughs. Soc. d'Exploitation de Brevets & d'Applications Industrielles (G.P. 434,997, 24.1.25. Conv., 4.2.24).—The sample of dough is placed between two plates, provided with openings which can be closed at will, and pressed to a thin disc. Air or gas under pressure is led into a chamber attached to one plate, so that the dough is blown out in the form of a bubble until it bursts. The work performed by the compressed air is recorded and is taken as a measure of the baking value of the flour.

F. R. Ennos.

Preparation of a baked product containing active Kephir bacilli. R. Stauffer (Swiss P. 115,303, 28.7.25).—The dough is mixed with milk in which fermentation due to the presence of Kephir bacilli has begun, allowed to ferment further, and then baked.

F. R. Ennos.

Process and apparatus for preserving or improving foodstuffs. P. St. G. Kirke (E.P. 265,416, 30.3.26).—Foodstuffs liable to fermentation or putrefaction are placed in a container with an opening, and inserted in a chamber which may be heated. The chamber is provided with pipes for evacuating the whole apparatus and introducing an inert gas, and also with a device, externally operated, for closing the opening in the container before removing the latter.

F. R. Ennos.

Treatment of flour and other milling products. J. van Loon (F.P. 609,057, 8.1.26).—The flour etc. is mixed with persalts, peroxides, or perozonides, together with crystalline substances such as quartz and calcium phosphate. The bleaching action of the per-compounds on the flour is thereby increased. F. R. Ennos.

Method and apparatus for the treatment of milk powder. A. E. White. From Merrell-Soule Co. (E.P. 265,323, 24.11.25).—The milk powder is packed in a suitable container, which is evacuated through an opening having an area not substantially greater than 0·0013 cm.^{2} per lb. of powder. After remaining under the vacuum for a period until the free or uncombined oxygen is reduced below 5 c.c./lb. of powder, the container is charged with carbon dioxide and sealed. F. R. Ennos.

Preparation of cheese of constant flavour and long-keeping quality. L'Auvergne Laitière (F.P. 608,615—6, 2.4.25).—The uncooked cheese is heated at 75° and the liquid portion poured into moulds and allowed to cool. To facilitate the melting of the cheese, a small quantity of a salt of a weak acid, *e.g.*, 2% of sodium tartrate, is added. F. R. Ennos.

Production of artificial mineral water. H. W. A. Branco (G.P. 432,472, 14.11.24).—The ash obtained by burning mixtures of charcoal from different sources is extracted with spring water or with water containing lime or silica in solution. L. A. Coles.

Production of beet molasses for feeding stuffs. E. Giron (F.P. 608,390, 24.12.25).—The finely-divided sugar beets are subjected to pressure and the expressed juice is purified, evaporated, and mixed with the pulp, which has been converted into dextrose by hydrolysis with hydrochloric acid and neutralisation of the acid with sodium carbonate. The resulting mixture is evaporated to the desired consistency. F. R. Ennos.

Preparation of a foodstuff. Brown Co. (Can. P. 259,391, 24.3.25).—After removal of the oil from peanuts by pressure, it is hydrogenated and mixed with peanut meal, prepared from the residue, and with milk powder. F. R. Ennos.

Malt product (E.P. 264,890).—See XVIII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Technical preparation of ferric saccharate and ferric ammonium citrate. F. Chemnitius (Chem.-Ztg., 1927, **51**, 121—122).—These two salts can be conveniently manufactured in connexion with solid ferric chloride, a solution of which is the raw material used. It is not necessary that sulphate or free chlorine should be absent. Ferric saccharate is prepared as follows:

Ferric chloride solution is mixed with hot soda solution in excess in wooden vessels fitted with agitators. The precipitated ferric hydroxide is washed by decantation and blown through a filter press. The pressed cake is placed in a jacketed, enamelled pan with sufficient distilled water to prevent any dehydration. To 30 kg. of paste 150 kg. of sugar are added and, when the mixture boils, 12 litres of 15% sodium hydroxide solution. The resulting dark brown syrup is heated for 6 hrs., run on to iron trays, and dried *in vacuo*. The iron content of the dried salt is determined and the material is then ground with addition of powdered sugar if required. The colour of the final preparation can be adjusted by addition of 0·5% of water. The yield on the sugar used is 97%. For the preparation of ferric ammonium citrate, ferric hydroxide prepared as above is used. For a content of 30 kg. of iron, 169 kg. of citric acid free from lead are used, and the mixture of hydroxide and citric acid solution is warmed in a jacketed, enamelled pan. When dissolution has begun, 30 kg. of hexamethylenetetramine are added, the liquid is filtered and evaporated to a syrup. If any ferrous iron is present it is oxidised by addition of hydrogen peroxide. The concentrate is brushed out on glass plates and dried at 60°, forming greenish-yellow scales. C. IRWIN.

Determination of the basic constant of morphine and its application in the titration of morphine. I. M. KOLTHOFF (Pharm. J., 1927, **118**, 126—127).—Polemical. Morton (B., 1926, 644) found from measurements of the hydrogen-ion concentration of morphine hydrochloride that at 30° the basic dissociation constant k_b of the base was $6·3\times10^{-9}$. The author (A., 1926, 125) found from measurements in mixtures of the base and its hydrochloride a value of $7·5\times10^{-7}$ at 15° (whence methyl red is the best indicator for the titration of the base or its salts with strong acids), whilst the acid dissociation constant k_a of the base was $1·4\times10^{-10}$. From the last two constants, the p_H of the base is about 9·0 at the isoelectric point. This point corresponds to the minimum solubility of morphine in water amounting at 18° to a concentration of 5×10^{-10} molar, or 147 mg./litre. B. FULLMAN.

Stability of atropine and hyoscyamine during analysis. S. PALKIN and H. R. WATKINS (J. Amer. Pharm. Assoc., 1927, **16**, 21—30. Cf. B., 1926, 963).—In the determination of atropine or hyoscyamine by extraction with chloroform or benzene and titration of the residue after removal of the solvent, the following precautions should be observed in view of the possible hydrolysis of these alkaloids. They should be first treated with water containing at least an equivalent and preferably excess of acid, it being then safe to heat in order to effect complete solution. For the liberation of the alkaloid with ammonia preparatory to extraction, the cooled aqueous solution should be shaken immediately with a solvent to remove the bulk of the alkaloid as rapidly as possible. The chloroform or benzene solution may then be evaporated on a steam bath to a low volume (5—10 c.c.), but not to dryness, after which 0·02*N*-sulphuric acid is added before removing the remainder of the solvent and titrating the residue with 0·02*N*-alkali (methyl red). If 20% of absolute alcohol is added to the chloroform solution, the latter may be evaporated to dryness without risk of hydrolysis of the alkaloids, which are unstable in water alone. Ammonia appears to exert much less activity as a hydrolytic agent than has been supposed. W. J. POWELL.

Assay of *Hyoscyamus*. A. STIKAROVSKY (J. Amer. Pharm. Assoc., 1927, **16**, 30—32).—Low results obtained in the determination of the alkaloid content of tincture of *Hyoscyamus* are due in the majority of cases to over-heating during evaporation of the chloroform solution. The dry alkaloid should on no account be exposed to heat, hence the last 3 c.c. of solvent are best removed at ordinary temperature in a current of air. W. J. POWELL.

Assay of salicylates and benzoates. II. A. H. CLARK (J. Amer. Pharm. Assoc., 1927, **16**, 18—21).—The method for the determination of salicylic or benzoic acid in medicinal salts, in which the acid is extracted from an aqueous solution of the salt with chloroform and the chloroform solution evaporated under standardised conditions (B., 1926, 767) is suitable for use with ammonium, bismuth, strontium, lithium, and basic mercury salicylates. Methyl salicylate is first boiled with a solution of sodium hydroxide in 50% alcohol, the alcohol removed by distillation, and the acid extracted with chloroform after acidification. Phenyl salicylate is hydrolysed in a similar manner, the alkaline liquid is saturated with carbon dioxide, extracted with chloroform to remove phenol, then acidified, and extracted again to obtain the salicylic acid. It is suggested that the purity of these substances is most conveniently controlled by a determination of their salicylic acid contents, and suitable standards are quoted. W. J. POWELL.

Determination of 2-phenylquinoline-4-carboxylic acid in tablets. L. E. WARREN (J. Amer. Pharm. Assoc., 1927, **16**, 32—41).—The solubility of 2-phenylquinoline-4-carboxylic acid in a number of solvents has been determined, the most satisfactory for its extraction from tablets being anhydrous ether, ethyl alcohol (95%), and acetone. Provided that the tablet contains no other acid, the substance can be rapidly determined by heating the pulverised tablet with neutral alcohol, and titrating the solution with 0·1*N*-sodium hydroxide (phenolphthalein). If acetylsalicylic acid or salicylic acid is present, satisfactory results may be obtained by precipitating and weighing the silver salt of 2-phenylquinoline-4-carboxylic acid. W. J. POWELL.

D.M. index for the chemical testing of arsenobenzenes. A. PATTA (Boll. Chim. farm., 1927, **66**, 65—67).—The author's previous conclusions (cf. B., 1925, 826) are upheld against the criticisms of De Myttenaere (cf. B., 1926, 385). T. H. POPE.

Rhizome and roots of *Podophyllum peltatum*, L. H. L. KUESTER (J. Amer. Pharm. Assoc., 1926, **15**, 259—263).—Values for the rhizomes and roots, respectively, are: water, 63—74, 52—74%; resin, 3·89 (by U.S.P. method 3·19), 5·16%; ash from resin, 5·67, 3·98%. Sucrose was present in the extracts.

CHEMICAL ABSTRACTS.

Ephedrine content of *Ephedra vulgaris*, var. *helvetica*. R. E. SCHOETZOV and G. H. NEEDHAM (J. Amer. Pharm. Assoc., 1926, **15**, 1070—1071).—Three samples of *Ephedra vulgaris*, var. *helvetica*, assayed by the U.S.P. IX method for belladonna root, using methyl red as indicator and the $0 \cdot 1N$-sulphuric acid factor of 0·016513 given by Chen (*ibid.*, 1925, **14**, 189) for ephedrine, gave 0·403%, 0·632%, and 0·863% of alkaloids calculated as ephedrine. These values compare with the amounts 0·305% and 0·515% found by Masucci and Suto (*ibid.*, 1926, **15**, 748), and are much higher than those reported by Chen of 0·018% and 0·091%. E. H. SHARPLES.

Preparation and examination of pine-needle extract. P. BOHRISCH (Pharm. Ztg., 1927, **72**, 278—281).—The methods of extraction are reviewed and criticised, and the following recommended. The young branches are distilled for several hours, allowed to cool, and again distilled. The residue is heated for several hours with water and, after cooling, the liquid is expressed, added to the two distillates from which the oil has been separated, and the whole evaporated down to a thin syrup. In the factory the oils separated by the first distillation are finally added to the aqueous extract after evaporation of the latter *in vacuo*. Adulteration with crude sulphite liquors is common; the extent of this is determined by distilling the sulphur dioxide from the extract acidified with phosphoric acid into a potassium iodide solution or over iodide-starch paper. Analytical results for a large number of different samples are given. S. I. LEVY.

Spanish spike lavender oil. Influence of steam-distillation. E. TEDESKO (Perf. Essent. Oil Rec., 1927, **18**, 44—45).—The wide variation in character of these oils is largely due to the methods used in their preparation and to the nature of the soil in which the plant is grown, and no influence can be ascribed to the altitude. The following figures are typical of genuine oils:—(1) Steam-distilled (Albocacer, 2500 ft.); d^{15} 0·9123, α +2·5°, alcohols (as borneol) 32·5%, cineole (Kleber and von Rechenberg's method) 35·4%, solubility in 70% alcohol 1 in 2·5 vols., in 65% alcohol 1 in 5 vols. (2) Distilled on open fire (Albocacer, 2500 ft.); d^{15} 0·9090, α +1·30°, alcohols 34·0%, cineole 33·0%, solubility in 70% alcohol 1 in 2 vols., in 65% alcohol 1 in 3·5 vols. (3) Distilled on open fire (Aguaviva, 1500 ft.); d^{15} 0·9046, α −2·35°, alcohols 38·5%, cineole 28·5%, solubility in 70% alcohol 1 in 1·5 vols., in 65% alcohol 1 in 3 vols. Steam-distilled oils are characterised by a higher sp. gr., a stronger tendency to dextrorotation, and a superior perfume value. E. H. SHARPLES.

French lavender oils. The ester question. I. P. LANGLAIS and J. GOBY. **II.** A. RECLAIRE (Perf. Essent. Oil Rec., 1927, **18**, 47–49).—I. The authors disagree with Parry (La Parfumerie Moderne, 1926, Dec.), who is of the opinion that linalyl butyrate is the predominating ester in lavender oil, and from a repetition of their previous experiments (B., 1927, 92) maintain that the acetic ester predominates, and that in a genuine lavender oil 94% of the free fatty acids liberated on acidification of the saponified oil consists of acetic acid. II. Evidence in support of the above. The odour of butyric acid has never been observed in the liberated acids from a large number of French lavender oils, and it is quite distinct in those from a synthetic oil containing 40% of linalyl butyrate. E. H. SHARPLES.

Detection and determination of lauric esters. M. S. SALAMON (Perf. Essent. Oil Rec., 1927, **18**, 59).—The following method is suitable for the detection and determination of esters of lauric and non-volatile fatty acids in lavender and bergamot oils. 10 c.c. of oil are saponified for 1 hr. with 12·5 c.c. of $2N$-potassium hydroxide, the mixture is transferred to a separating funnel, and after the addition of 50 c.c. of water, shaken with 25 c.c. of ether. The aqueous layer is again extracted with 25 c.c. of ether, the ether extracts are mixed and given three washes with water each of about 10 c.c. The washings are added to the main aqueous solution, which is now acidified, twice extracted with 25 c.c. of ether, again washed with water to remove water-soluble acids, and evaporated. A little absolute alcohol is added and evaporated off, and the residue dried to constant weight. Pure bergamot oils yield not more than 2·5% of residue, whilst lavender and similar oils yield less than 1%. From genuine oils the residue is sticky and resinous, and its solution in aqueous soda gives only a light brown precipitate on the addition of barium chloride solution. The residue from adulterated oils, if allowed to cool, usually becomes crystalline, and with the alkaline solution barium chloride gives a heavy, white, curdy precipitate which can be filtered off and the acid separated (cf. also B., 1919, 553). E. H. SHARPLES.

Reaction for eugenol. C. J. ENKLAAR (Chem. Weekblad, 1927, **24**, 115—116).—A freshly-prepared solution of manganic sulphate containing acetic acid, when shaken with ethereal oils containing eugenol, gives a red oily precipitate soluble in benzene. The red oil contains manganese and carbon; on boiling or when cold it gives a yellowish solid. S. I. LEVY.

Rapid reaction to distinguish anise oil from star anise oil. W. P. H. VAN DEN D. MAREEUW (Pharm. Weekblad, 1927, **64**, 189—195).—The reactions serving to identify cineole, which is a constituent of star anise oil, but not of anise oil, have been examined, and it is found that the ferrocyanide and ferricyanide tests are suitable. If star anise oil is shaken with acid solutions of these reagents, crystalline precipitates are obtained; anise oil gives at most an opalescence. S. I. LEVY.

Detection of fungus poison. AUFRECHT (Chem.-Ztg., 1927, **51**, 130).—The poisonous constituent (helvellic acid) is isolated from the fungus previously dried below 60° by extraction with alcohol, formation of the soluble barium salt, liberation of the acid, and subsequent purification with alcohol, and finally with ether. The acid is a yellow syrup soluble in water, alcohol, and ether, and contains no nitrogen or sulphur. The aqueous solution shows strong reducing properties, and gives no precipitate with the ordinary alkaloid reagents. It is unstable, and loses its power by heating with alkali, or by keeping at room temperature for 3 days. F. R. ENNOS.

PATENTS.

Manufacture of acetic acid. H. DREYFUS (E.P. 264,558, 13.6.25. Cf. B., 1926, 1013; 1927, 125).—Acetic acid is formed when equimolar mixtures of methyl alcohol and carbon monoxide (or mixtures containing excess of the latter) are heated below 350—450° (preferably 200—300°) under pressures up to above 200 atm. (generally 50—150) in the presence of catalysts capable of forming acetates which decompose with the formation of acetic acid below 400—450° (preferably 200—300°), such as the oxide of copper, tin, lead, or zinc; copper or zinc acetate; zinc, aluminium, or tin methoxide, etc., either alone or mixed with one another, or with potassium or sodium acetate or methoxide. Operation is continuous, with regeneration of the catalyst. (Reference is directed in pursuance of Sect. 8, Sub-sect. 2, of the Patents and Designs Acts, 1907 and 1919, to E.P. 254,819 [B., 1926, 721]).

Making formaldehyde from methylene chloride. E. KRAUSE and K. RÓKA, Assrs. to HOLZVERKOHLUNGS-IND. A.-G. (U.S.P. 1,616,533, 8.2.27. Appl., 1.9.22. Conv., 17.9.21).—Methylene chloride is heated in the presence of water in a closed vessel at 140—170°.

A. DAVIDSON.

Preparation of maleic and succinic acids from furfuraldehyde. ZAIDAN HOJIN RIKAGAKU KENKYUJO, Assees. of T. YABUTA (E.P. 253,877, 18.5.26. Conv., 22.6.25).—Furfuraldehyde is subjected to electrolytic oxidation. *E.g.*, in a cell of two compartments, separated by a porous diaphragm, the anodic compartment contains 5% sulphuric acid, in which furfuraldehyde is dissolved or suspended, and the cathodic 10% sulphuric acid. The electrodes are lead pipe coils, which serve also as coolers. A low current density of about 0·02 amp./cm.^{2} is used, and the temperature kept at 35°. Electrolysis is continued until the anolyte no longer gives the reaction of furfuraldehyde with aniline acetate. It is then moderately concentrated and, on cooling, mixed crystals of succinic and maleic acids (80% and 20% respectively) separate. If succinic acid alone is required, the current is reversed after the aldehyde has been completely oxidised, when the maleic acid is reduced. Using a current density of 0·06 amp./cm.^{2} with a lead peroxide anode, the product is chiefly maleic acid in about 75% yield. In the above examples an increased yield is obtained by the addition of vanadium, cerium, or manganese compounds as catalysts in the anolyte.

A. DAVIDSON.

Production of pyrrolidine derivatives. E. C. BRITTON, Assr. to DOW CHEMICAL CO. (U.S.P. 1,607,605, 23.11.26. Appl., 1.10.25. Cf. Löffler and Freytag; A., 1909, i, 830).—A secondary aliphatic amine in which a methyl group is separated by a chain of 3 carbon atoms from the nitrogen atom, and which contains no group, other than the amino-group, reactive under the conditions of the synthesis, is treated under cooling with a solution of 1·5 g.-mol. of sodium hypochlorite or hypobromite, and the *N*-halogeno-derivative, which separates, is heated *in vacuo* at 145° with sulphuric or phosphoric acid, when the corresponding 1-pyrrolidine derivative is obtained. *E.g.*, di-*n*-butylamine yields 1-n-*butylpyrrolidine*, b.p. 151°.

T. S. WHEELER.

Production of arseno-bismuth compound. G. W. RAIZISS and A. KREMENS, Assrs. to ABBOTT LABORATORIES (U.S.P. 1,605,691, 2.11.26. Appl., 15.10.25).—An alkali bismuth tartrate, *e.g.*, potassium bismuth tartrate, is mixed in aqueous solution with the disodium salt of 3 : 3′-diamino-4 : 4′-dihydroxyarsenobenzene, or similar compound, and the liquid is poured into a mixture of ether and methyl alcohol, when a *product*, with the general formula R·As : Bi·As(R)·Bi : As·R, where R is an aminoaryl radical, precipitates. It is non-toxic, and is of value in the treatment of spirochætal infections.

T. S. WHEELER.

Making allium preparations for medical use. CHEM. FABR. VORM. SANDOZ (E.P. 235,883, 16.6.25. Conv., 17.6.24).—The odour and irritating effects of the allium species are removed by treating the comminuted product with animal or vegetable charcoal and drying in air, or *in vacuo* at low temperature. B. FULLMAN.

Method of manufacturing from garlic an injection for tuberculosis. K. KUBOTA (E.P. 264,960, 14.11.25). —The bulbous roots of wild or cultivated garlic (*Allium sativum*) are dry-distilled at 100°, rising to 600°. The product on filtration yields a liquid, *d* 1·01—1·017, suitable for injections. B. FULLMAN.

Producing tuberculosis protective and curative material. H. LANGER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,617,060, 8.2.27. Appl., 28.9.25. Conv., 21.8.24).—Prolific cultures of tubercle bacilli are produced in a culture medium containing 1 pt. of methylene blue in about 300,000 pts. of the medium.

A. DAVIDSON.

Production of anthrax aggression. H. JENSEN, C. E. SALSBERY, and G. G. GRAHAM (U.S.P. 1,615,876, 1.2.27. Appl., 19.7.26).

Laxative chewing gum. HEALTH PRODUCTS CORP., Assees. of A. H. COURT (E.P. 249,816, 17.12.25. Conv., 25.3.25).

Auro-sodium thiosulphate solutions (E.P. 246,809). —See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Intensification of the latent image on photographic plates and films. I. Latent image intensification. E. P. WIGHTMAN and R. F. QUIRK (J. Franklin Inst., 1927, **203**, 261—287).—Hydrogen peroxide and silver nitrate, and probably other noble metal salts, increase the developability of a plate above its normal value, if applied between exposure and development. "Intensification" of the exposed portions is greater than that of the unexposed parts. A strong intensification is obtained with a concentration low enough not to produce appreciable fog. The effect is approximately equivalent in degree to that produced by a brief flash exposure to light before or after the main exposure. With the same concentration of peroxide and time of treatment, intensification was greater on the fast plates used than on medium-speed plates, and no

intensification resulted on process plates, Eastman portrait film, N.C. film, and plates desensitised with chromic acid. By increasing either the peroxide concentration or time of treatment a certain intensification could be obtained on these materials. With desensitised plates and no light exposure, no more than a slight latent fog could be produced with any peroxide concentration or time of treatment. Water gives an apparent intensification of the latent image which is ascribed partly to swelling of the gelatin, allowing a more rapid penetration of the developer. The ratio of gamma of peroxide-treated to untreated plates increases with increasing time of development. Latent image intensification in general increases somewhat with increasing development time when measured as a density effect, and decreases when measured as an H. and D. speed difference. In absence of other intensifying agents and oxidisers, 1 pt. of peroxide in 10 million pts. of water can be determined to within 20—30% by the latent-image intensification method. The view is supported that light produces a developable and an undevelopable image, and that peroxide makes the latter image developable in producing latent-image intensification. There is less undevelopable image formed by light in medium and slow-speed and desensitised plates, and consequently the same concentration of peroxide and time of treatment gives different degrees of intensification on plates of different speed. W. CLARK.

Fogging action of persulphate. W. CLARK (Brit. J. Phot., 1927, 74, 121—122).—The density of fogging by persulphate increases with increasing time of treatment, but soon reaches a maximum at a very low density value, and then decreases with increasing time of action. The action is by no means so pronounced as that of hydrogen peroxide. Acidified persulphate gives a somewhat more pronounced effect than the plain solution. The reversal effect is probably due to a competitive action between the fogging tendency of persulphate and its destructive action on the latent image. W. CLARK.

PATENTS.

Manufacture of silver halide emulsion. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (E.P. 255,846, 8.7.26).—In the manufacture of silver halide emulsions, water-soluble salts containing at least three atoms of sulphur, selenium, or tellurium are added either to the emulsion or to its constituents. Suitable salts are sodium trithionate, sodium tetrathionate, or the sodium salt of trithiodiglycollic acid, or the corresponding selenium and tellurium salts. An increase of sensitiveness without fog results, and gelatins normally unsuited for photographic purposes are rendered useful. W. CLARK.

Producing photographic papers for making prints from tracings. H. KAMMERER (U.S.P. 1,618,931, 22.2.27. Appl., 14.6.26. Conv., 7.5.25).—See E.P. 252,012; B., 1927, 93.

Imprinting photographic plates, films, and the like. J. WOLF (E.P. 254,295, 15.6.26. Conv., 29.6.25).

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

[Non-solvent, propulsive] explosive. BOMBRINI PARODI-DELFINO, and T. BENELLI (E.P. 265,029, 24.3.26). —Non-solvent, propulsive powders, low in nitroglycerin, of reduced corroding effect, and free from exudation, efflorescence, and crystallisation, are prepared by introduction into the powder of aromatic nitro-derivatives and centralites in eutectic proportions. Suitable proportions are: nitroglycerin, 5—35%; nitrocellulose, 60—70%; dinitrotoluene, 3—8% (or a mixture of di- and tri-nitrotoluene, 12—25%); and diphenyldiethylcarbamide, 1—10%. S. BINNING.

Match-striking composition. H. W. ROBINSON (E.P. 265,378, 4.2.26 and 4.11.26).—The usual materials, such as red phosphorus, antimony sulphide, and an abrasive, are mixed with materials for forming a condensate gum, and the condensation is carried out by heating the mixture to 100—130° in a steam autoclave, the time required being 2 hrs. at the higher temperature. The materials for forming the condensate gum may be a phenol, a formaldehyde, and an exciter. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Chemical characteristics of sewage sludge. S. L. NEAVE and A. M. BUSWELL (Ind. Eng. Chem., 1927, **19**, 233—234).—Fresh and digested sludges cannot be distinguished either by volatile matter and fixed carbon determinations, or by differences in grease content, but there is a marked difference in the pyridine-soluble humic acid content, which definitely increases during sludge digestion. Determinations of the loss in weight of dry degreased sludge when extracted for 18 hrs. by buffered solutions of varying p_H values (6·2—9·8) at 25°, show that the rate of peptisation of sludge increases with increase of p_H up to p_H 7·4, after which the curve is relatively flat. W. T. LOCKETT.

Practical application of hydrogen-ion control in the digestion of sewage sludge. S. E. COBURN (Ind. Eng. Chem., 1927, **19**, 235—236).—According to Rudolfs and Baity, favourable digestion takes place at p_H 7·3 to 7·6. At Plainfield, Conn., low temperatures during the early months of operation, and the presence of considerable amounts of grain mash which tended to produce acid digestion, were apparently the causes for the failure of an Imhoff tank to produce well-digested sludge for two years. Seeding with small amounts of well-digested sludge produced no satisfactory results. Treatment with hydrated lime in order to neutralise the acidity was then tried. By sprinkling the scum with lime at intervals and working it in with a long-handled shovel, the p_H of the scum and sludge was raised from 6·3 to 7·3 in the course of a few months, and favourable digestion commenced. The lime treatment was then stopped. With the approach of summer weather both sludge and scum continued to improve without the aid of lime. Later, by accident, nearly the whole of the sludge was run off, when acid digestion began again with rapid accumulation of offensive scum. By treatment of the scum with lime as before favourable digestion was re-established. W. T. LOCKETT.

Effect of temperature on sewage sludge digestion. W. RUDOLFS (Ind. Eng. Chem., 1927, **19**, 241—243).—Digestion of unseeded fresh solids (raw sludge) is extremely slow at temperatures below 10°. Raising the temperature slightly above 10° has comparatively little effect. Digestion time is materially decreased with higher temperatures. Maximum digestion takes place at about 27—28°. The addition of lime sufficient to produce a slightly alkaline reaction of the medium (p_H 7·3—7·6) decreases digestion time still further. Definite quantities of organic material produce about the same total volume of gas at all temperatures from 10° to 35°. The volume of gas can be increased and its composition changed by changing the reaction of the medium. This is due to the development of organisms of a different type. In a given time the average number of bacteria per g. of organic matter in unadjusted sludge does not increase with increase in temperature, whereas the average numbers of bacteria in lime-treated sludge decrease with the increase in temperature. In the same given time protozoan numbers (mainly small flagellates) follow the bacterial numbers. W. T. LOCKETT.

Rôle of iron in the activated-sludge process. A. WOLMAN (Eng. News-Rec., 1927, **98**, 202—204).—Some theoretical and practical foundation for assuming that iron may be helpful to the process, both as an oxygen carrier and as an absorbent and coagulant, is furnished by the recent experiments of Warburg (A., 1925, 1481), and those of Horowitz-Wlassowa (Z. Hygiene u. Infektions-Kranheit, 1926, **105**, 98), which indicate that the coagulating properties of activated sludge are due mainly to the presence of iron and aluminium hydronides. Studies of the rôle of iron in nitrate production in soils and other oxidation processes in nature, and the results of a superficial survey of a large number of activated sludge plants, from which it would appear that the plants which give least difficulty in operation deal with sewages which contain appreciable quantities of iron, give further support to the hypothesis, but the hypothetical and experimental data so far available are inadequate to demonstrate finally its validity. W. T. LOCKETT.

Behaviour of active chlorine preparations towards organic materials. E. REMY (Biochem. Z., 1927, **180**, 97—104).—The following antiseptics are placed in decreasing order of their ease of decomposition by organic matter: antiformin (alkaline sodium hypochlorite), hypochlorite (aqueous solution of sodium hypochlorite), caporite (calcium hypochlorite), multisept (mixture of equimolecular amounts of succinic acid and bleaching powder), pantosept (sodium dichlorosulphonamidobenzoic acid), chloramine (sodium *p*-toluenesulphonchloroamide), and magnocide (basic magnesium hypochlorite). P. W. CLUTTERBUCK.

Disposal of trade waste from a plant mercerising, bleaching, and dyeing cotton materials. W. E. HADLEY (Ind. Eng. Chem., 1927, **19**, 239—241).—Before discharge into the central collection tanks the alkaline waste liquors from the mercerising plant are brought near to neutrality by treatment with sulphuric acid, and when considered necessary the colour of the waste dye liquors while in a concentrated form is destroyed by addition of bleach liquor. The nearly neutral composite waste from all departments is treated with hydrated lime and aluminium sulphate. W. T. LOCKETT.

Chemical treatment of trade waste. II. Wastes from silk dyeing. F. D. SNELL and D. S. BRUCE (Ind. Eng. Chem., 1927, **19**, 237—239).—The plant supplying the data given is a relatively small piece-dye works dyeing silk and silk-cotton goods principally with direct dyes. In addition to dye wastes there are others, *e.g.*, mercerising liquors, silk and silk-cotton boil-offs, and bleach liquors, at more or less frequent intervals. The plant is subject to rulings by which it is compelled to treat 90% of the waste in such a way as to be harmless to fish and plant life when discharged into a river; permission is given to discharge the remainder into the sanitary sewer. Silk boil-off, rich in soap, is treated with acid for by-product recovery. Silk-cotton boil-off is diverted to the sewer. Mercerising liquors are neutralised and mixed with the dye-waste. Other miscellaneous liquors are mixed with the dye waste without pre-treatment. A half-hour average of the combined dye and other wastes (oxygen consumed 100—400 p.p.m.) treated with 5 lb. of copperas and 4 lb. of lime per 1000 gals. settles satisfactorily. Decolorisation is better with an hour average. Acceptable results are obtained with 1 hr. average and 4 hrs. settlement. The effluent when undiluted is only slightly toxic to fish life; the sludge is discharged into the sewer. W. T. LOCKETT.

Comparative chemical results of slow and rapid filtration [of public water supplies]. F. EGGER (Chem.-Ztg., 1927, **51**, 94).—Clarification with aluminium sulphate, followed by rapid filtration, is compared with slow sand filtration in the case of river and lake water forming the Stuttgart supply. No advantage was found in adding more than 20 mg. of aluminium sulphate per litre, and 2½ hrs. was allowed for flocculation. The oxygen absorption was reduced by 40%—rather better than with sand filtration; the chlorine figures were similar in the two methods. The bacterial counts in the former method are less satisfactory and chlorination is advisable. Free carbon dioxide, present in the soft lake water, interferes with flocculation, but can be removed by agitation. The taste is considered to be better with the rapid filtration treatment. C. IRWIN.

PATENTS.

Purification of water. ÉTABL. PHILLIPS & PAIN (F.P. 608,171, 28.3.25).—A mixture of water with part of the solution of sodium compounds from a base-exchanging purifying plant is treated with lime to precipitate carbonates and bicarbonates. L. A. COLES.

Process and apparatus for the removal of oil from condensation waters. J. B. GAIL and N. ADAM (U.S.P. 1,617,208, 8.2.27. Appl., 11.4.21. Conv., 5.4.20).—See E.P. 180,420; B., 1922, 565 A.

Clarifying and purifying liquids and waste waters. A. J. RAVNESTAD (U.S.P. 1,619,036, 1.3.27. Appl., 26.10.23. Conv., 28.9.20).—See E.P. 225,635; B., 1925, 113.

Protective coating for water tubes (E.P. 260,233).—See I.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 22, 1927.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Hydrogenation of lignite in the presence of aqueous bicarbonate solution. F. Fischer and A. Jäger (Abh. Kenntn. Kohle, 1925, 7, 141—143; Chem. Zentr., 1926, II, 1915).—Hydrogenation by the action of hydrogen under pressure in the presence of aqueous sodium bicarbonate solution is preferable to the use of sodium formate or mixtures of carbon monoxide and steam. L. A. Coles.

Colorimetric determination of humus substances. U. Springer (Brennstoff-Chem., 1927, 8, 17—20).—The colour intensity of solutions of humic acids in aqueous alkalis (*e.g.*, sodium hydroxide and carbonate, ammonia, and, to a less degree, pyridine) decreases on keeping, rapidly at first and then more gradually. This decomposition effect increases with increasing concentration and degree of dissociation of the base.
W. T. K. Braunholtz.

Methane fermentation of peat. E. Melin, S. Norrbin, and S. Odén (Ingeniörs Vetenskapsakad. Handlingar, 1926, [53], 1—42).—Fermentation of unheated peat with inoculating sludge from sedimentation and decomposition chambers (Imhoff tanks) proceeded very slowly. The rate of fermentation was considerable only when the bacteria were vigorously stimulated at the start. The rate of fermentation was greatest at 25—30°, and at p_H 6·5—7. Addition of 0·05—1% of calcium acetate increased the rate of fermentation considerably. Analyses of the gases evolved during fermentation showed 62—78% CH_4 and 30—13% H. The constituents of the peat which seemed to be fermented were the hydrocelluloses and mucilage substances, hexoxanes in general, cellulose, and to some extent the pentosans. No indication of attack of the humic acids or lignins was found. Experiments on the removal of water from the fermented peat by pressure indicated that the water could not be squeezed out of fermented peat any more readily than from raw peat. Obviously, the colloids were not destroyed by the micro-organisms. W. Clark.

Recovery of carbon dioxide from waste flue gases. M. A. Rabinovitch (J. Russ. Phys. Chem. Soc., 1926, 58, 518—526).—To ascertain the best conditions for the recovery of carbon dioxide from waste flue gases, the absorption and subsequent evolution of carbon dioxide by potassium hydroxide absorbers was investigated. The gas was passed at various pressures (1—8 atm.) and temperatures (19—80°) through potassium hydroxide solutions, and the rate of adsorption, and its relation to the concentrations of potassium carbonate and potassium hydrogen carbonate, as also the rate of their decomposition and recovery of the gas, were determined. Both rates increase with rise of temperature. Increase of potassium hydrogen carbonate lowers the rate of absorption. The dissociation pressure decreases with rise in potassium carbonate concentration. Experiments with mixtures of air and carbon dioxide were also carried out. The equation

$$[KHCO_3]^2\{[K_2CO_3]^2 \times P_{CO_2}\} = K$$

holds over a considerable range of concentrations. Addition of small quantities of potassium sulphate, which is formed in practice from the sulphur dioxide present in the flue gases, decreased the efficiency of the absorbers; potassium chloride had the same effect. Traces of soluble calcium or magnesium salts restored the efficiency almost to its original level. M. Zvegintzov.

Thermal relations in a Scottish oil-shale retort. L. C. Karrick (Chem. Met. Eng., 1926, 33, 740—744).—In the average Scottish oil-shale retort, which is 26 in. in diam. in the distilling zone, the importance of steam and water-gas as internal heating media is indicated. It is shown that 55% of the heat necessary to distil the shale is transferred from the hot zone to the low-temperature distillation zone by these fluid heat-carriers, and that 86% of this heat is recovered from the spent shale. If the size of shale particles is reduced from 4 in. to 1 in. it is possible to increase the capacity of the retort by the use of larger quantities of steam which reduce the temperature in the hot zone to below that required for water-gas production. This conserves heat within the retort, but necessitates the provision of external means for generating fuel gas for heating the retort setting.
S. Pexton.

Petroleum oils of Grozny. A. Sachanen-Sachanov (Petroleum, 1926, 22, 1240—1244).—Crude oil rich in wax is found in the deeper layers of the new oilfield. It has *d* 0·840, solidif. pt. 15°, greatly affected by hysteresis, contains 7% of wax (m.p. 53—54°), and 0·25% S. The yield of benzine is 23·31%, and of kerosene 13·31%. The residue after removal of lubricating oils contains 9% of wax (m.p. 75°). Oil poor in wax is found in the Solenaja Balka field; it has *d* 0·840, solidif. pt. —10°, and yields 27·79% of benzine and 12·54% of kerosene. Oil free from wax (containing 0·1—0·5% only) is found in almost all horizons of the old field and in the highest layer of the new field. It has *d* 0·850—0·860, but at the refineries 0·870 owing to weathering; solidif. pt. below —20°, and contains 0·2—0·3% S. The yield of benzine is 28·53% and of kerosene 12·55%. The residue contains 18·9% of asphaltenes. The natural gases of Grozny have a temperature of 80—90°, and are generally of *d* 1·30—1·40.

They yield 1 kg. of benzine per cub. m., consisting of 30—40% of condensable hydrocarbons and 60—70% of methane, ethane, and propane. H. Moore.

Valuation of commercial benzine by the "index number" of Ostwald. E. Kroch (Petroleum, 1926, **22**, 1245—1247).—The index number is not a new conception, being represented in American practice by the average b.p. It is misleading as an indication of the commercial value of spirits, as is shown by distillation graphs of various benzines. H. Moore.

Resinification value of transformer oils. F. Hoeg (Petroleum, 1927, **23**, 116—117).—The author confirms Typke's suggestion (cf. B., 1926, 523) that in examining transformer oils, the resin content should be determined both before and after heating; of two oils having the same resin content after heating, that which has the higher content before heating, and consequently shows the smaller increase, would probably prove the better in use. The acid value of the oils increases to about 2·5 times its original value during heating, and is approximately proportional to the resinification value. L. A. Coles.

Deleterious properties of lubricating oils. J. E. Hackford (Oil Eng. Tech., 1926, **7**, 325—327; Chem. Abstr., 1926, **20**, 3561).—Air is bubbled (1 per sec.) through the oil at 150° for 9 hrs.; the rate of acidity formation is the difference between the total acidity (c.c. of 0·1*N*-potassium hydroxide per 10 g. of oil) and the inherent acidity. To determine the acidity which will cause damage to bearings, 50 c.c. of oil are extracted with boiling distilled water for 1 hr., the solution is filtered, poured to a definite mark in a 50 c.c. U-tube, and the voltage obtained between zinc and copper electrodes is measured. A. A. Eldridge.

Determination of small quantities of water in mineral oils. W. Boller (Petroleum, 1927, **23**, 146—148).—The method serves for the determination of water in transformer and other oils containing up to about 0·01% of water. A current of a purified and dried non-reacting gas, preferably hydrogen, is passed successively through a vessel containing the oil at 130—140°, a tube packed with calcium carbide, and an absorption vessel containing an ammoniacal solution of copper sulphate and hydroxylamine hydrochloride, After the hydrogen has been passed through the apparatus for 1 hr., the carbide tube is heated at 180—200° for 1 hr., and the precipitated copper acetylide is washed, dissolved in dilute sulphuric acid, precipitated as sulphide, ignited, and weighed as cupric oxide, or is dissolved in excess of acidified ferric sulphate solution, and the reduced iron titrated with permanganate. L. A. Coles.

Determination of paraffin scale in crude paraffin wax. E. Belani (Petroleum, 1926, **22**, 1347—1348).—A small fireclay crucible, thoroughly porous, glazed on the sides but unglazed at the bottom, is inverted on a cork, through which a tube communicates with a vacuum flask. By the use of gelatin a space is moulded on the bottom of the crucible, and is filled with wax mass. The adherent oil is drawn into the flask through the porous base of the crucible, the oil removed, and the amount of scale determined by difference. H. Moore.

Dopes and detonation. H. L. Callendar (Engineering, 1927, **123**, 147—148, 182—184, 210—212; cf. B., 1926, 618).—It appears probable from the nuclear theory of detonation that the reactions occurring in the cylinder during compression, but before ignition, determine the "knocking" or "anti-knocking" character of a fuel. The temperatures of initial combustion (T.I.C.) of a number of organic liquids have been determined by passing a mixture of the vapour and air through a glass tube, the temperature of which could be gradually raised; the T.I.C. was shown by the appearance of water, carbon dioxide, aldehydes, or acids. The T.I.C. of the aromatic hydrocarbons were higher than those of the alcohols (benzene, 670°; ethyl alcohol, to aldehyde, 445°; to carbon dioxide, 520°), and the latter higher than those of the paraffins (about 300° for pentane and hexane). The mixture strength, if above 10%, had no effect on the T.I.C. of benzene or alcohol, but the values for pentane and hexane passed through minima. The T.I.C. of detonating fuels, such as the paraffins or ether, are much lower than the recorded values of their ignition temperatures. The temperatures reached in the cylinder during compression are sufficiently high to initiate oxidation of the detonating fuels, but not to produce any appreciable action with other fuels. Increasing the engine speed diminishes the time available for such chemical changes to occur, and a corresponding variation of the highest useful compression ratio (H.U.C.R.) with speed is observed. The addition of small quantities of anti-detonating substances to detonating fuels raises the T.I.C. (as determined in the heated tube) sufficiently to prevent initial oxidation in the cylinder during compression; thus 1% of iron carbonyl raised the T.I.C. of undecane by 150°. The products of the initial combustion of hexane are principally aldehydes, with smaller quantities of carbon dioxide, water, carbon monoxide, ethylene, and other unsaturated substances etc. Addition of lead tetraethyl, iron carbonyl, or nickel carbonyl reduces very considerably the amount of oxidation, and entirely inhibits the formation of unsaturated substances. Some metallic surfaces, *e.g.*, iron, copper, lead, and, in particular, platinum black, lower the T.I.C. and diminish aldehyde formation; others, *e.g.*, tin, nickel, zinc, produce little effect. Alcohols are much less readily oxidised than the paraffins, and addition of dopes increases the amount of oxidation. The aldehydes which appear as oxidation products of the paraffins are not therefore produced by the intermediate formation of alcohols. Moreover, the formation of aldehydes cannot be regarded as a primary cause of detonation, direct addition of aldehydes in fact making possible a small increase in the H.U.C.R. Examination of the products of initial oxidation of the paraffins does not support the theory of the intermediate formation of hydroxyl compounds, but indicates that the first products of oxidation are peroxides. Peroxide formation occurs in the oxidation of ethers and paraffins, but not with alcohols, benzene, or phenols. Addition of dope completely inhibits peroxide formation. Small additions

of organic peroxides to fuels have a marked effect in promoting detonation. It is concluded that detonation is due to the accumulation of peroxides in the nuclear drops during rapid compression. The amount of peroxide is not sufficient in itself to cause the detonation observed, but acts as a primer, causing simultaneous ignition of the drops. The metallic dopes act by reducing the peroxides as fast as they are formed, and thus delay the ignition of the drops. A. B. Manning.

Patents.

Manufacture of gas. H. Nielsen and B. Laing (E.P. 262,834, 20.6.25).—Semi-coke produced by the low-temperature distillation of coals in a hot, gaseous heating medium is very reactive towards carbon dioxide, and can be utilised in a pulverulent form to reduce the carbon dioxide of industrial heating gases to carbon monoxide, thereby producing higher-grade gas of lower sp. gr. A combined plant for the process is described. The gases containing carbon dioxide from the distillation retort are stripped of tar and oils and preheated at 750—800° in one of two chequered chambers, the other meanwhile being raised to the desired temperature by the combustion of solid or gaseous fuel. The hot gases from the preheater are brought into contact with the semi-coke, which may be heated externally as well as by the hot gases, the carbon dioxide being thereby reduced. The resulting hot gases may be utilised for the internal heating of the low-temperature distillation retort. If gas of still higher calorific value is required, part of the distillation products may be led direct from the retort to the preheating chequers prior to the reduction of its carbon dioxide content, and the tar oils contained therein cracked. S. Pexton.

Purifying [coal] gases. J. B. Garner, R. W. Miller, and G. A. Shaner, Assrs. to Standard Development Co. (U.S.P. 1,609,872, 7.12.26. Appl., 30.12.22).—Coal or like gas containing sulphur and cyanogen compounds, and other impurities, is washed with dilute (1—7%) sodium hydroxide solution, containing 0·5—4% of oleic acid, or a soluble soap, or other soluble substance, which increases the tendency to film formation, and hence the interaction of the gas and liquid. The liquid is revivified by treatment with ferric oxide, which is regenerated by exposure to air. Alternatively, ferric, copper, or nickel sulphate (1—10%), or one of the corresponding hydroxides, is added initially to the alkaline film-forming liquor, which is then regenerated, when necessary, by aeration. T. S. Wheeler.

Continuous operation of retorts for the production of gas and coke. Stettiner Chamotte-Fabr. A.-G. vorm. Didier (G.P. 434,086, 27.3.24).—A part of the coal is preheated to a temperature just above the softening point before being fed into the retort. "Hanging" of the charge in the retort is thereby avoided. A. B. Manning.

Cracking coal, oils, and other hydrocarbons. Internat. Bergin-Comp. voor Olie- en Kolen-Chemie, Assees. of A. Debo (Can. P. 258,201, 15.5.25).—The starting material is introduced, in the form of a paste, by means of a screw conveyor, into a high-pressure container, in which it is heated in the presence of hydrogen, the products of the reaction being withdrawn from the container in such a way as to maintain a constant level therein. A. B. Manning.

Cracking of [hydrocarbon] oils. F. E. Wellman, Assr. to Kansas City Gasoline Co. (U.S.P. 1,615,779, 25.1.27. Appl., 10.7.22).—The oil is contained in several small stills connected together; these are externally heated intermittently in periodic rotation, the resulting vapours being drawn off into a central vessel, while a common liquid level with a comparatively large surface is maintained in the small stills to prevent foaming. W. N. Hoyte.

Production of hydrocarbons resembling natural petroleum. L. Paris-Durcy (F.P. 607,688, 23.3.25).—Bituminous coal, lignite, tar, heavy oils, or vegetable or animal oils are hydrogenated by heating with hydrogen or other hydrogenating gas to pressures between 200 and 800 kg. A. B. Manning.

[Hydrocarbon] vapour recovery. C. L. Voress and N. C. Turner, Assrs. to Gasoline Recovery Corp. (U.S.P. 1,616,242, 1.2.27. Appl., 14.6.26).—The gaseous mixture of hydrocarbons is treated by contact with a solid adsorbent for an insufficient time to let the selective action of the adsorbent displace a large proportion of the more volatile vapours with less volatile vapours. A substantial part of the vapours is volatilised from this adsorbent, cooled, and passed through a second adsorbent in which the full selective action is permitted. A substantial part of the adsorbed vapours from this second adsorber is volatilised and condensed.

W. N. Hoyte.

Refining of [hydrocarbon] oils. E. B. Cobb, Assr. to Standard Development Co. (U.S.P. 1,616,352—3, 1.2.27. Appl., [a] 8.4.22, [b] 24.2.26).—(a) Sulpho-compounds, which induce emulsification and are present in oils treated with sulphuric acid, are destroyed by maintaining the oils at 180—260° until such compounds are decomposed. (b) Heavy distillates are treated with large amounts of fuming sulphuric acid, and the oil is neutralised and distilled by direct heat and by steam to a 5—10% residue. The distillate is re-treated with a less proportion of acid, and the resulting oil maintained at 180—260° while steam is passed through until it becomes neutral and will not tarnish copper. It is finally filtered. W. N. Hoyte.

Separation of [liquid] hydrocarbons from mineral matter. M. Mallet (F.P. 607,269, 12.3.25).—Mineral matter containing oil is ground under water and a part of the oil is separated. The material then undergoes further mechanical treatment, during which the oil collects in drops on the surface of the water while the mineral matter sinks to the bottom. A. B. Manning.

Continuous distillation of oil. F. A. Howard and N. E. Loomis, Assrs. to Standard Development Co. (U.S.P. 1,613,754, 11.1.27. Appl., 15.6.22).—The oil is passed through a battery of stills heated externally; the vapours evolved from each still are passed through heat exchangers immersed in the preceding still; the vapours from each still are mixed with those from the succeeding still entering the heat exchanger.

W. N. Hoyte.

Distillation of oil. C. W. Stratford (U.S.P. 1,613,298, 4.1.27. Appl., 19.8.25).—The oil is contained in a still heated externally at the required temperature; a quantity is continuously drawn off at the bottom and returned to the still under pressure, being discharged through a large number of small nozzles so that the streams impinge on the heated sides of the still.
W. N. Hoyte.

Distillation of oil. J. Primrose, Assr. to Power Specialty Co. (U.S.P. 1,614,689, 18.1.27. Appl., 7.4.21). —The vapours evolved from a conventional still are passed through a series of air-cooled condensers, the resulting condensates are separately collected, and the hot air from the condensers is passed to the still furnace.
W. N. Hoyte.

Cracking of oils. H. H. Armstrong (U.S.P. 1,613,010, 4.1.27. Appl., 9.7.23).—The oil to be cracked is heated almost to cracking temperature and passed as a fine spray into the reaction chamber; a light hydrocarbon is also heated to cracking temperature, and is expanded into the atomised heavy oil so as to maintain the reaction chamber at the desired temperature.
W. N. Hoyte.

Cracking of oils. C. Owens (U.S.P. 1,613,124, 4.1.27. Appl., 28.5.21. Renewed 1.4.26).—The cracking stock is vaporised by a current of steam drawn through the oil; the mixture of steam and oil vapours is passed through a heating zone, and the products are subsequently condensed, the whole process being operated at atmospheric pressure. W. N. Hoyte.

Cracking of hydrocarbon oils. C. L. Parmelee, Assr. to Sinclair Refining Co. (U.S.P. 1,613,718, 11.1.27. Appl., 8.10.24).—The oil is heated to cracking temperature in a tube still, thence it passes to a separating chamber in which carbon and tarry matter are drawn off; the clarified liquid passes to the supply chamber which acts as a heat exchanger with the separating chamber. Vapours from both the chambers pass up a dephlegmator, and thence to a condenser. The runback from the dephlegmator together with fresh cracking stock is returned to the supply chamber, whence the oil is pumped to the cracking still. W. N. Hoyte.

Cracking [hydrocarbon] oils. O. P. Amend (U.S.P. 1,613,735, 11.1.27. Appl., 3.3.22).—The oil contained in a still is heated to cracking temperature by electric heating elements immersed in it. A minimum pressure of 75 lb. is maintained on the still. W. N. Hoyte.

Cracking of hydrocarbons. A. J. Paris, jun. (U.S.P. 1,614,930, 18.1.27. Appl., 14.5.24).—The oil is distilled and the distillate is heated to cracking temperature under pressure, the boiling range of the fraction so treated being limited to less than 50°. W. N. Hoyte.

Process of cracking or treating oils and other similar hydrocarbons. H. T. Darlington and R. M. Steffen, Assrs. to M. B. Schuster (U.S.P. 1,614,660, 18.1.27. Appl., 2.6.23).—Hydrocarbons under conditions of temperature and pressure conducive to cracking are treated with a gel composed of bentonite and water.
M. E. Nottage.

Production of liquid resembling petroleum. P. J. H. Baurier (F.P. 606,900, 25.2.25).—Tar, oil, or gases obtained by the distillation of carbonaceous material are treated with steam at 300—450° in the presence of metals capable of decomposing the steam.
L. A. Coles.

Producing natural gas gasoline. F. E. Hosmer (U.S.P. 1,613,108, 4.1.27. Appl., 16.7.26).—The natural gas is compressed and cooled by spraying with a cooling medium which is chilled by cold gas obtained on releasing the pressure on the gas at a later stage in the plant.
W. N. Hoyte.

Motor fuel. C. F. Kettering and T. Midgley, jun., Assrs. to General Motors Corp. (U.S.P. 1,605,663—4, 2.11.26. Appl.,[a],15.4.22. Conv., 7.5.19. [b], 3.5.24).—The fuel comprises a low-compression motor fuel and a compound (a) in which at least one univalent hydrocarbon radical, or (b) having more than one aromatic closed chain and in which at least one aromatic hydrocarbon radical is substituted for a hydrogen atom of ammonia. H. Moore.

Flash-point tester for oils and fuels. H. Jentzsch (G.P. 433,432, 12.2.25).—An ignition block which can be heated in a furnace has a receptacle in which are a number of borings for holding the substance to be investigated, and is provided also with a multiple-flame burner for igniting the substance. The results, which are much more accurate than those obtained with the known open crucible type of apparatus, agree with those given by a closed apparatus. A. B. Manning.

Motor-fuel-testing composition. V. Serravaoll and E. Weis (U.S.P. 1,615,143, 18.1.27. Appl., 16.8.23. Conv., 21.12.22).—The testing fluid consists of a mixture of phenol and amyl acetate, containing two differently-coloured dyes, only one of which is soluble in petrol, tetralin, and benzine. The colours of the layers formed when the fuel under test is mixed with the liquid give an indication of the composition of the fuel, as regards alcohol and benzene on the one hand, and petrol, tetralin, and benzine on the other.
T. S. Wheeler.

Hydrocarbon fuel. Combustible fuel [for internal-combustion engines]. L. Kirschbraun (U.S.P. 1,614,559—60, 18.1.27. Appl., [a] 1.4.20, [b] 16.8.20. Renewed [a] 8.6.25, [b] 9.7.26).—(a) Naphthenic acid or a naphthenesulphonic acid (1 pt.) is dissolved in petrol (100 pts.), the mixture is heated above 100° under pressure, dry steam (10 pts.) is passed into it, and the whole is homogenised and cooled. The product is a stable emulsion. (b) Petrol (50 pts.), the sulphonic acid from the acid sludge obtained in oil-refining (50 pts.), and water (20 pts.) are mixed with petrol to give a product containing 0·5% of sulphonic acid, 6% of water, and the remainder petroleum. Both products are non-carbonising. T. S. Wheeler.

Motor spirit. J. Luciani (F.P. 608,186, 28.8.25).—Various alcohols, nitroglycerin, tetranitrotoluene, or a mixture of these is added to benzene or its derivatives.
A. B. Manning.

IV.—DYESTUFFS AND INTERMEDIATES.

Patents.

Vat dyes. I. G. Farbenind. A.-G., Assees. of G. Kränzlein, H. Greune, and R. Sedlmayr (G.P. 430,558, 21.8.24; Addn. to 412,053).—Benzanthrone or one of its

derivatives containing at least one free *peri*-position is condensed with aromatic *o*-dicarboxylic anhydrides in the presence of aluminium chloride or other condensing agent. The dye from benzanthrone and phthalic anhydride dyes cotton yellow from the hyposulphite vat; similar products are obtained from α-hydroxybenzanthrone with phthalic anhydride, and from β-methylbenzanthrone with 3 : 6-dichlorophthalic anhydride.

C. Hollins.

Benzanthrone derivatives. I. G. Farbenind. A.-G., Assees. of H. Neresheimer (G.P. 431,744, 17.8.22).—Benzanthrones having a free 2-position are treated with acid oxidising agents. Benzanthrone is converted by chromic and sulphuric acids into a *sulphate*, which, by boiling with water, gives an oxidation *product*, convertible by sulphonation into a yellow wool *dye*. Chlorobenzanthronecarboxylic acid gives with sulphuric acid and manganese dioxide an oxidation *product*, which may be purified by treatment with sodium hypochlorite and precipitation with acids. C. Hollins.

***iso*Dibenzanthrone.** I. G. Farbenind. A.-G., Assees. of H. Neresheimer (G.P. 431,775, 26.10.24).—3-Chlorobenzanthrone, when treated with alkaline hydroxides and alkoxides in an indifferent diluent, *e.g.*, with potassium hydroxide, *iso*propyl alcohol, and benzene at 10—15°, yields pure *iso*dibenzanthrone. C. Hollins.

[*Bz*-2¹-]Hydroxybenzanthrones. K. Schirmacher, K. Zahn, K. Wilke, and P. Ochwat, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,614,398, 11.1.27. Appl., 28.4.24. Conv., 25.6.23; cf. E.P. 218,255 and 224,522; B., 1925, 538,583).—*Bz*-1¹-Nitrobenzanthrone is reduced to the amino-compound, which is acetylated with acetyl chloride, and treated with concentrated nitric acid at 50°, when nitration occurs in the *Bz*-2¹-position. The nitroamino-compound, obtained by hydrolysis on treatment with nitrosylsulphuric acid gives a diazohydroxy-anhydride, the nitro-group being eliminated as nitrous acid, which, on treatment with stannous chloride in alkaline solution gives Bz-2¹-*hydroxybenzanthrone*, m.p. above 280°; Bz-2¹-*acetoxybenzanthrone* has m.p. 201°. Bz-2¹-*Methoxybenzanthrone*, m.p. 171—173°, on heating with alcoholic potassium hydroxide solution gives a *dye* which yields fast bluish-green shades on cotton in a hyposulphite vat. Bz-2¹-*Ethoxybenzanthrone* has m.p. 169—170°. These compounds can also be obtained by heating *o*-alkoxyphenyl α-naphthyl ketones with aluminium chloride. T. S. Wheeler.

Manufacture of polynitroamines. W. H. Bentley, and W. Blyth & Co., Ltd. (E.P. 263,552, 2.10.25).—Halogen atoms in aromatic polynitrohalogen compounds are replaced by amino-groups by heating the halogen compounds with carbamide (or with an organic substance which produces carbamide or is produced from it during the reaction, *e.g.*, biuret, cyanuric acid, or ammonium cyanate), in an open vessel, at 130—150°, preferably with agitation, if necessary in the presence of an inert solvent. Examples are given of the preparation, using carbamide, of 2 : 4-dinitroaniline from 1-chloro-2 : 4-dinitrobenzene; 4-chloro-2 : 6-dinitroaniline from 1 : 4-dichloro-2 : 6-dinitrobenzene; and 2 : 4 : 5 : 7-tetranitro-α-naphthylamine from 1-bromo-2 : 4 : 5 : 7-tetranitronaphthalene. B. Fullman.

Manufacture of water-insoluble azo dyes. Farbenfabr. vorm. F. Bayer & Co. (F.P. 604,443, 12.10.25).—The diazo compounds of dyes obtained by coupling diazotised unsulphonated amines with sulphamic acids of aromatic amines are coupled in substance or on the fibre with second components containing no sulpho- or carboxyl groups, such as β-naphthol, arylides of 2 : 3-hydroxynaphthoic acid, or acetoacetic acid, pyrazolones, etc. Thus, by coupling diazotised *o*-anisidine in alkaline solution with α-naphthylsulphamic acid, diazotising the product, and immersing in the clear diazo solution a fabric padded with 2 : 3-hydroxynaphthoic-β-naphthylamide, black shades are obtained. Using in place of the last-named substance 1-phenyl-3-methyl-5-pyrazolone scarlet shades are formed, and with α-naphthol-4-carboxylic acid black shades. A. Davidson.

Manufacture of azo dyes. Chem. Fabr. Griesheim-Elektron (F.P. 601,709, 6.8.25. Conv., 16.8.24).—Sulphonated or unsulphonated diazo, tetrazo, or diazoazo compounds are coupled with arylides of 2-naphthol-6-carboxylic acid. The dyes can be formed on the fibre, whilst those prepared in substance can be used as wool dyes or for the preparation of lakes etc. From diazotised 4-chloro-2-nitroaniline and the anilide of 2-naphthol-6-carboxylic acid a light-fast, orange-red lake is obtained. Cotton padded with bis-2 : 6-hydroxynaphthoic-1 : 6-naphthylenediamide and developed with diazotised 2 : 5-dichloroaniline yields fast brown shades. The azo dye from diazotised aniline-2 : 4-disulphonic acid and bis-2 : 6-hydroxynaphthoic dianisidide dyes wool fast orange shades. A. Davidson.

Preparation of halogenated naphthasultones. I. G. Farbenind. A.-G., Assees. of K. Schirmacher and W. Langbein (G.P. 433,527, 5.8.24).—α-Naphthol-4 : 8- or -6 : 8-disulphonic acid is heated in presence of water with halogens or halogenating agents. No naphthaquinones are formed, and the products, which are used as dyestuff intermediates, are converted by alkalis into halogenated naphtholsulphonic acids. *E.g.*, an aqueous solution of the sodium salt of α-naphthol-4 : 8-disulphonic acid is treated with hydrochloric acid and the mixture saturated with chlorine at about 30°; more chlorine is introduced, and the mixture heated at 80° for 30 min. On cooling, 4-*chloro*-1 : 8-*naphthasultone*, m.p. 176—178°, crystallises. 4-*Bromo*-1 : 8-*naphthasultone*, m.p. 196—198°, and *sodium* 4-*chloro*-1 : 8-*naphthasultone*-6-*sulphonic acid* are described.

E. H. Sharples.

Azo dyes. I. G. Farbenind. A.-G., Assees. of L. Laska and A. Zitscher (G.P. 430,580, 4.10.24).—Azo dyes, suitable especially as ice-colours, are made by coupling amino-*N*-arylbenz- or-naphtha-triazoles with arylamides of 2 : 3-hydroxynaphthoic acid. 2-o-, 2-m-, and 2-p-*Aminophenyl*-1 : 2-*naphthatriazoles* melt at 120°, 160°, and 203—205°, respectively; 2-(3-*amino*-4-*methylphenyl*)-1 : 2-*naphthatriazole* at 172—173°; 2-(3-*amino*-4-*methoxyphenyl*)-1 : 2-*naphthatriazole* at 185°; 2-(2-*amino*-4-*methylphenyl*)-1 : 2-*naphthatriazole* at 137°; and 2-(4-*chloro*-2-*aminophenyl*)-1 : 2-*naphthatriazole* at 192°. The dyeings are fast to bowking. C. Hollins.

Preparation of mercaptans of the naphthalene series. I. G. Farbenind. A.-G., Assees. of J. Haller

(G.P. 433,103, 24.2.25).—Diazo compounds of naphthylaminesulphonic acids by treatment with alkali sulphides or xanthates are converted into dinaphthyldisulphidedisulphonic acids which are reduced to mercaptans, the sulpho-group being removed either during or before the reduction. E. H. SHARPLES.

Preparation of 6-chloro-2-nitrotoluene-4-sulphonic acid and 6-chloro-o-toluidine-4-sulphonic acid. I. G. FARBENIND. A.-G., Assees. of F. HENLE and B. VOSSEN (G.P. 434,402, 2.9.24).—(1) 6-Chloro-2-nitrotoluene is sulphonated with fuming sulphuric acid; the sulphonic acid is separated as the sodium salt by treatment with sodium chloride and reduced to 6-chloro-*o*-toluidine-4-sulphonic acid. (2) The technical mixture of 6-chloro-*o*-toluidine and 4-chloro-2-nitrotoluene is sulphonated. (3) The unchanged 4-chloro-2-nitrotoluene is separated by the addition of weak sodium chloride solution in which the 6-chloro-derivative is soluble, and then the latter is salted out as its sodium salt by the addition of more sodium chloride. The oxidation of 6-chloro-2-nitrotoluene-4-sulphonic acid to 6-chloro-2-nitro-4-sulphobenzene-1-carboxylic acid and the preparation from the latter of the amino-derivative are described. E. H. SHARPLES.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of isoelectric points of wool and silk fibroïn. L. MEUNIER and G. REY (Compt. rend., 1927, **184**, 285—287).—Curves relating the percentage swelling with the p_H value of the solution in which this occurs have been constructed for wool and silk fibroïn. The degree of swelling was found by deducting the weight of the dry threads from that of the centrifuged swollen threads. Minimum values of the swelling are found for p_H values of 3·6—3·8 and 4·2 in the case of wool and silk fibroïn, respectively, the former being the more pronounced. This indicates that wool is more amphoteric in nature than silk, since the swelling of proteins is due to the formation of ionisable salts with acids and bases. This is confirmed by determinations of the amino-nitrogen by Van Slyke's method, mean values of 0·2% and 0·07% being found in wool and silk, respectively. Shrinkage is a minimum in solutions, the p_H value of which corresponds to the isoelectric point of the protein.

J. GRANT.

Methods of studying cord tyre fabric. F. W. STAVELY and N. A. SHEPARD (Ind. Eng. Chem., 1927, **19**, 296—301).—Fatigue in tyre cords is measured by hysteresis loss and flexing life, the former showing that the properties of the original cord are not maintained, and the latter being of value in determining the desirability of changes in cord construction. When well impregnated with rubber cements containing vulcanisers etc. (the solvents of which should readily wet the fibre) each strand of a cord is completely covered with rubber, and the flexing life is improved. Increased flexing life usually corresponds with increased mileage. The effect of factors such as humidity, temperature, and wax content in the testing of raw cords is emphasised. The removal of the acetone- and benzene-soluble constituents of the cotton cord lowered the flexing life by about 30%.

C. O HARVEY.

Testing sulphite liquor. E. SCHMIDT (Zellstoff u. Papier, 1927, **7**, 56—57).—The determination of free sulphurous acid in sulphite cooking liquor by the potassium iodate method is not more accurate than by titration with sodium hydroxide and phenolphthalein. The latter method breaks down owing to the sensitiveness of the indicator to carbon dioxide and organic acids, but the former also measures the total acidity (due to sulphurous acid, carbon dioxide, and organic acids). The effect of traces of either carbon dioxide or organic acid added to potassium iodate–iodide solution is to cause liberation of iodine. Excess of water partially reverses the above reaction, as shown by experiments in which various quantities of water free from carbon dioxide were added to solutions of potassium iodate and iodide acidified with equal volumes of standard acid, and the mixtures titrated with sodium thiosulphate solution, the required volume of the latter diminishing as the quantity of added water increased. B. P. RIDGE.

Recovery of condensed water in cellulose factories. R. ESCOURROU (Chim. et Ind., 1927, **17**, 21—24).—By condensing the steam from the heating tubes at just below the b.p. and using this water for further steam raising, a great economy in fuel is effected, but it is essential to ensure that there is no contamination by acid leaking into the heating tubes. For this purpose two electrodes, immersed in the stream of condensing water, are connected through an electric lamp, relay, and bell to a source of electric supply. When the condensed water is pure no current flows through the circuit, but the presence of acid increases its conductivity and causes the lamp and bell to function

F. R. ENNOS.

Chlorine gas process in the paper industry. G. CONSIGLIO (Papier-Fabr., 1926, **24**, 785—789).—Particulars are given of a factory at Naples where cellulose is prepared from esparto, straw, and hemp by the chlorine gas process operated in conjunction with an electrolytic caustic soda plant. The grass is first softened by treatment in open boilers with a 1—2% solution of caustic soda at 70—80°. The alkali solution is used repeatedly, being made up in strength and volume by the addition of fresh liquor. Soda solution and wash waters are circulated by pumps, the steeped grass being washed in the boilers. After washing, the grass is transported to the chlorinating chambers of concrete. The chlorinated material is first washed with water and then extracted with cold caustic soda solution. The other processes are as usual. The consumption of chemicals per ton of bleached esparto cellulose is caustic soda 160—180 kg., chlorine gas 400 kg., chlorine in the form of bleach liquor 20 kg. The process suitably modified has also been applied experimentally to the manufacture of cellulose from bagasse and from pine wood. J. F. BRIGGS.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Ice-colour process. I. G. FARBENIND. A.-G., Assees. of W. CHRIST (G.P. 433,149, 2.12.24).—Improved fastness to rubbing is obtained in ice-colours by treating the impregnated material, with or without previous centrifuging, pressing, or draining, with a weaker impregnating

solution to which sodium hydroxide and an electrolyte have been added, or with a solution of an electrolyte containing a little sodium hydroxide, or with dilute sodium hydroxide solution alone, in all cases with or without the addition of formaldehyde. The process is applicable to ice-colours made from 2 : 3-hydroxynaphthoyl- or acetoacetyl-derivatives of arylamines and arylenediamines. C. Hollins.

Preparation of colour reserves with vat dyes under vat dyes. A. Lauterbach, and Gebr. Enderlin Druckfabr. u. Mech. Weberei A.-G. (Austr. P. 103,911, 10.6.25).—To the previously prepared goods one or more vat dyes are applied with neutral metallic compounds as reserves, and dyed, it may be after steaming, in a dye bath of a vat dye, when simultaneously the reserving of the dye by the dye bath takes place. The dye to be reserved can also be applied by cover-printing or padding, and then after-treated with an alkaline solution containing, if necessary, a reducing agent. As reserves, there may be used substances of alkaline reaction or reducing agents or both. A fabric is printed with a colour reserve of lead acetate, potassium carbonate, gum senegal, kaolin, and Indanthrene Yellow G, dried, and then dyed, without steaming, by a hyposulphite vat of Indanthrene Blue RS. It is then washed, soured, washed, and soaped at the boil, a yellow under blue being obtained. A. Davidson.

Developing salts [in dyeing]. I. G. Farbenind. A.-G. Assees of H. Krzikalla (G.P. 433,150—1, [a] 12.4.25, [b] 29.4.25. Addns. to 421,837; B., 1926, 318).—In the preparation of anhydrous mixtures containing a nitrite and the mineral acid salts of aromatic amines capable of yielding a diazo solution when dissolved in water as claimed in the chief patent, the mineral acid salts are replaced by (a) *N*-sulphonic acid derivatives of diazotisable amines or (b) salts other than mineral acid salts of diazotisable amines; *e.g.*, a satisfactory developing mixture comprises (a) Glauber's salt, sodium bisulphate, sodium nitrite, and the sodium salt of a naphthalenesulphoamino-compound, or (b) acid aniline oxalate (or aniline salts of phthalic or aromatic sulphonic acids, *e.g.*, benzenesulphonic acid), Glauber's salts, oxalic acid, and sodium nitrite. A. J. Hall.

Dyeing and printing cellulose esters. I. G. Farbenind. A.-G., Assees. of R. Metzger and C. Schuster (G.P. 433,349, 11.10.24).—Cellulose acetate etc. is dyed with azo dyes derived from aminoaldehydes. *p*-Aminobenzaldehyde, diazotised and coupled with salicylic acid, gives a yellow; with 1 : 7-aminonaphthol, maroon; with β-naphthol, subsequently treated on the fibre with phenylhydrazine, brown. *m*-Aminobenzaldehyde, diazotised and coupled with dimethylaniline gives a golden-orange; with diphenylamine a golden-yellow. C. Hollins.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Macroscopical determination of carbon in Martin steel test pieces. M. Hamasumi (Sci. Rep. Tôhoku Imp. Univ., 1926, **15**, 819—828).—In the acid process the final decarburisation is so slow that samples may be withdrawn and analysed chemically, but in the basic process a quick macroscopic method is essential. Specimens of typical steels with carbon contents from 0·1 to 0·9% were quenched from 850° and 900° and the hardness was measured with a Rockwell testing machine and plotted graphically against the composition. The angles through which the specimens could be bent without fracture were also measured, and the etching effects with picric acid are illustrated, and also the different types of fracture. All the above properties are suited for the approximate determination of carbon, but the accuracy from 0—0·3% carbon is much greater than that above 0·3%. The macrostructures are also illustrated, and the effects discussed from the point of view of the equilibrium diagram. W. Hume-Rothery.

Hardness of different structures in steel. K. Tamaru (Sci. Rep. Tôhoku Imp. Univ., 1926, **15**, 829—850).—The hardness of the different constituents has been measured for both plain carbon and manganese steels. A carbon steel containing 1·69% C was heat-treated and quenched so as to give structures containing varying amounts of austenite and martensite, the hardness of which was found to be 155 and 720 respectively (Brinell scale), assuming that the hardness varies linearly with the proportion of the constituents. The impact hardness of 0·6% and 0·8% carbon steels was measured between 0° and 900°; both show slight minima at 400—500°, and maxima at 600°, above which the hardness falls rapidly. The hardness of the austenite in manganese steels diminishes as the carbon content increases from 0·1—0·6%, and also diminishes with increasing manganese content, but from 0·6—0·9% C the hardness (182) is independent both of carbon and manganese contents. These steels contained 12—20% Mn, and a little martensite may have been present; they were also examined at high temperatures and showed complex results. In martensite produced by the ordinary quenching of carbon steels the hardness shows a maximum at about 0·8% C, but such specimens always contain some γ-iron. The hardness of a quenched 0·89% carbon steel is increased by tempering at 112°, but higher temperatures cause a decrease. The natural hardness of cementite is about 620, but in cast specimens it may be as high as 820 owing to strain. W. Hume-Rothery.

Change in hardness and density of iron and steel on cold-working. T. Ishigaki (Sci. Rep. Tôhoku Imp. Univ., 1926, **15**, 777—794).—The hardness and density of Armco iron and of steels of varying carbon content have been measured after various kinds of deformation. In specimens broken under tension, the hardness at different points along the test piece increases as the fracture is approached. In hammered specimens, no further increase in hardness occurs after the thickness has been reduced by 50%. Hydrostatic pressure causes a comparatively slight but quite definite increase in hardness. On tempering deformed specimens of Armco iron, the hardness increases up to 250°, then decreases until 470°, when a second slight hardening occurs between 470° and 560°, above which the material rapidly softens. Steels show a similar increase in hardness up to 300—400°. The decrease in density of specimens after deformation

has been measured. The densities of specimens which had been compressed in steel moulds were slightly less than those of the originals, but the pressure-density curves were complex. The magnetic changes produced on heating deformed specimens were studied, and the results are discussed theoretically.

W. Hume-Rothery.

Solubility of carbon in pure iron. Y. Yamada (Sci. Rep. Tôhoku Imp. Univ., 1926, **15**, 851—855).—The electrical conductivities of specimens of iron containing 0·010—0·089% C have been measured; the impurities present were 0·008% S, 0·002% Si, and a trace of manganese. The continuous nature of the composition-conductivity curves indicates that the solubility of carbon in pure α-iron is less than 0·01%, and this is confirmed by micro-examination.

W. Hume-Rothery.

Dilatometric and magnetic researches on pure iron and on iron-carbon alloys. H. Esser (Stahl u. Eisen, 1927, **47**, 337—344).—From a review of the work of previous investigators, and from the fact that only a very slight change of volume takes place at the A2 point of iron, the author concludes that the A2 change in iron is not a true allotropic modification, and that β-iron is really only a special condition of α-iron over a certain temperature range associated with a change in magnetic properties. The peculiar hook-shaped deviation obtained at the Ar1 point in the temperature-differential expansion curve of steels also occurs in that of pure electrolytic iron, and cannot, therefore, be caused by a retardation of the pearlite separation, but is probably due to temperature differences between the test and standard bars. Dilatometric analysis of pure iron that has been de-gassed by repeated heating at 700—800° *in vacuo* places the Ar3 point at 898° and the Ac3 at 904°; the Ac1 point is detectable only when at least 0·06% C is present in a steel, and the Ar1 when there is 0·20% C. With 0·86% C both the A1 and A3 points coincide, the Ar points at 708°, and the Ac points at 736°. The line joining the Ac1 points of iron-carbon alloys containing up to 0·86% C is horizontal at 736°, whereas that joining the Ar1 points rises in a smooth curve from 685° with 0·1% C to 708° with 0·86% C. The Ar3 and Ac3 lines fall steeply in smooth curves to join the A1 lines at the eutectoid point. Magnetic tests on a hypereutectoid steel (1·2% C) showed that the ratio, length/diameter, of the test piece had an appreciable effect on the cementite transformation at 180—200°.

A. R. Powell.

Rational use of case-hardening compounds. Systematic case-hardening tests. J. Hébert (Techn. moderne, 1926, **18**, 481—491, 525—532; Chem. Abstr., 1926, **20**, 3428).—The case-hardening effect of wood charcoal alone increases with the temperature; the rate of variation in the carbon content from the surface inwards decreases with increase of time, and increases with fall of case-hardening temperature. Penetration of 1·40 mm. is effected at 950° in 3 hrs. Addition of sodium chloride, sodium carbonate, ammonium chloride (10%), pulverised bone, or bone-black retards, and that of potassium ferrocyanide (10%), rosin, barium carbonate, carbonised leather, or sodium hydrogen carbonate accelerates the action, the character of the effects being described. With 20% instead of 10%, sodium chloride and potassium ferrocyanide increased the depth of penetration; carbonised leather, pulverised bone, and bone-black increased the outer eutectic or hypereutectic layer; rosin, barium carbonate, and ammonium chloride reduced both the total depth of penetration and the outer zone of maximum carburisation. The formation of an outer hypereutectic zone can be prevented by carrying out the treatment in two stages: (*a*) at 900—950° until the depth of penetration is 50—75% of that required, and (*b*) at 760—780°. It is important to obtain a sufficiently thick outer eutectic zone, and an inner, transition, hypoeutectic zone thick enough to reduce to a minimum the danger of fissuring on quenching.

A. A. Eldridge.

Cementation of iron, nickel, and cobalt by means of boron. Feszczenko-Czopowse (Trav. ac. mines Cracovie, 1925, [5]; Chem. Abstr., 1926, **20**, 3429).—Tests were carried out on "normal" mild steel (0·075% C), "abnormal" mild steel (0·115% C, 0·197% O), hypoeutectic steel (0·4% C), hypereutectic steel (0·95% C), nickel, cobalt, nickel steel (5 and 25% Ni), and nickel-chromium steels (0·5% Cr, 2·5% Ni; 1·12% Cr, 4·2% Ni). In gases containing carbon, cementation by carbon and by boron takes place simultaneously; boronisation is successful in hydrogen or *in vacuo*. The boronisation of iron and steel progresses very irregularly. At high temperatures the superficial layer cemented by boron is a solid solution of boron and carbon in γ-iron; on cooling, the alloy assumes a eutectoid structure. The rate of diffusion of boron in iron and steel rapidly increases with the temperature, but the layer so obtained is porous, and may be removed by slight mechanical effort. Iron is deoxidised by boron. The mechanism of the diffusion of boron in iron is considered. Boronisation of nickel takes place more readily than that of iron. Giebenhausen's diagram should include the solid solution of boron in α-nickel. Boron also gives a crystalline solid solution in α-cobalt. With greater boronisation of nickel a new, easily-fusible eutectic is formed. The rate of boronisation of cobalt is intermediate between those of iron and nickel. Nickel steels and nickel-chromium steels are more rapidly cemented with boron than mild steels, and the latter more rapidly than steels of higher carbon content.

A. A. Eldridge.

Reduction of iron ores by hydrogen. I. G. Gallo (Annali Chim. Appl., 1927, **17**, 27—38).—The action of hydrogen on magnetite of the percentage composition, Fe_3O_4, 86·53; SiO_2, 4·12; MgO, 7·20; MnO, 0·32; Al_2O_3, 0·71; S, 0·008; CoO, 0·10; CaO, 0·86; P, 0·002, and on the same ore after torrefaction and conglomeration, when it contained 86·85% Fe_2O_3, is described. The apparatus used was free from rubber and ebonite joints, as these are attacked by the hydrogen at a comparatively low temperature, with liberation of hydrogen sulphide. Reduction of the natural magnetite by pure hydrogen begins at 180°, and that of the torrefied product at 220°, but as the temperature rises (to 550°) the action becomes more rapid with the latter than with the former. After the first point of arrest in the manometer used to follow the change—this point being different for the two materials—a second is observed at

340—360°, which apparently marks the commencement of the reduction of the ferrous oxide. If water vapour as well as hydrogen is present, initiation of the reduction is retarded and, for the torrefied material, occurs at about 280—290°. The second stage, the reduction of the ferrous oxide, is also retarded and begins at 370° (375—380°) if the partial pressure of the water vapour is 20 (92) mm.; the velocity of the reduction also is lowered considerably by the presence of water vapour.

T. H. Pope.

Behaviour of iron pyrites with hydrogen. II. G. Gallo (Annali Chim. Appl., 1927, 17, 39—44).—The desulphuration of pyrites by means of hydrogen takes place in three successive stages: (1) $2FeS_2 + H_2 = Fe_2S_3 + H_2S$; (2) $Fe_2S_3 + H_2 = 2FeS + H_2S$; and (3) $FeS + H_2 = Fe + H_2S$, the temperatures at which these commence being respectively 228—230°, 280—285°, and 370—375°. The reaction may be arrested at the completion of phase (1) if the temperature is maintained for some time at 450°. But, although this and phase (2) may be completed in a short time, phase (3) requires longer for its completion, as well as a temperature appreciably higher than that at which it begins. This indicates that the heat of formation of ferrous sulphide from iron and sulphur differs widely from that of pyrites from ferrous sulphide and sulphur. The reversible reaction (3) is intensely endothermic, but if the temperature is raised gradually, the desulphuration of ferrous sulphide may be carried to completion. No sulphur compound analogous to Fe_3O_4 appears to exist.

T. H. Pope.

Anomalous internal friction of reversible ferro-nickels. P. Chevenard (Compt. rend., 1927, 184, 378—380).—A three-dimensional diagram has been constructed to show the variation of the decrease in reversible ferro-nickels in iron–nickel alloys as a function of the temperature and of the nickel content. The decrease corresponds to the internal friction of the ferro-nickels. A marked anomaly in the decrease is shown up to 300° for alloys containing 30—60% Ni, a maximum being obtained for 38% Ni. The internal friction is sensitive to secondary influences, such as the state of oxidation, and the thermal and mechanical histories of the sample. In terms of the author's theory (A., 1921, ii, 484) of a progressive and reversible transformation of an α form stable at absolute zero to an isomorphic β form stable to heat, the α form possesses a greater internal friction than the β form. The reaction $\alpha \rightarrow \beta$ occurs in the neighbourhood of the alloy Fe_2Ni, accompanied by condensation and by an increase in the modulus of elasticity.

J. Grant.

Magnetic analysis as a means of studying the structure of non-magnetic alloys. K. Honda and H. Endo (Inst. Metals, March, 1927, advance copy, 21 pp. Cf. B., 1918, 586 A).—Magnetic analysis can be applied to the study of the structure of paramagnetic and diamagnetic elements and alloys in both the solid and the liquid phases. A detailed description of the apparatus used for measuring magnetic susceptibility is given. In the case of the elements, the magnetic susceptibility–temperature curves are continuous except during melting or allotropic transformation. With binary alloys forming an eutectic, the susceptibility–concentration curve is linear in the range of the eutectic mixture and bends generally in the region of solid solution; the solution of a small quantity of a feebly diamagnetic or paramagnetic element in a strongly diamagnetic element in the solid phase decreases the diamagnetic susceptibility of the latter to a marked degree. Where no eutectic is formed, and the two components form a continuous series of solid solutions with one another, and also in the liquid phase, where the structure is a true solution, the susceptibility–concentration curve is linear, or nearly so. The magnetic susceptibility of an intermetallic compound has a special characteristic value quite different from the susceptibilities of the components. Magnetic susceptibility measurements show that intermetallic compounds existing in the solid state up to the liquidus may also persist in the liquid phase, although in some cases the compounds are more or less dissociated; the degree of dissociation can be roughly determined. Magnetic analysis also gives satisfactory results for the solidus and solubility line of cementite in the iron–carbon system, which is difficult to determine by thermal analysis. M. E. Nottage.

Examination of a fifteenth-century "brass." J. N. Friend and W. E. Thorneycroft (Inst. Metals, March, 1927, advance copy, 2 pp.).—Fragments from an English ecclesiastical brass which was cast in 1453 have been found to contain 84·1% Cu, 8·2% Zn, 3·6% Sn, 2·6%Fe, and 1·2% Pb. Microscopical examination in the unetched state clearly reveals the presence of lead particles, and the etched structure shows the typical cored configuration of cast bronze. M. Cook.

Penetration of brass by tin and solder. Copper-tin equilibrium diagram. H. J. Miller (Inst. Metals, March, 1927, advance copy, 11 pp.).—The results of experiments upon the straining of brass test-pieces when surrounded by molten tin, lead, and lead–tin solder, show that the stress necessary to cause cracking and penetration is much greater than that leading to penetration by mercury. Penetration usually takes place between grain boundaries, and the phenomenon is associated with season-cracking. The cracking of brass and subsequent penetration by the solder is due primarily to the presence of stress, and no cases of cracking are noted in the absence of stress. Liability of brass to crack during tinning or soldering is overcome by low-temperature annealing to remove residual injurious stresses. Evidence obtained from annealing experiments and microscopic examination indicate that the eutectic at the tin end of the copper–tin series of alloys contains between 0·6 and 0·7% Cu, which figure is lower than that obtained by previous investigators. M. Cook.

Attack of molten metals on non-ferrous metals and alloys. H. J. Hartley (Inst. Metals, March, 1927, advance copy, 22 pp.).—Clean polished surfaces of brasses and bronze were exposed to the action of molten tin at 300°. With bronze (91% Cu) and α or β brass, there is no sign of selective attack at the grain boundaries. With $\alpha\beta$ and $\beta\gamma$ brasses, the phase of higher zinc content is the more resistant, but the $\beta\gamma$ brasses show signs of fairly deep penetration along the grain boundaries. Prolonged attack upon $\alpha\beta$ and $\beta\gamma$ brasses results in the formation of a brittle constituent contain-

ing approximately 43% Cu, 45% Zn, and 12% Sn, which, on the grounds of *X*-ray crystal analysis, is regarded as a solid solution of the two intermetallic compounds Cu_5Zn_8 and Cu_4Sn. Tensile tests were carried out on brass and copper wires immersed in molten tin, or solders containing tin, at 240°, the times required for fracture under different stresses being measured. Solution and penetration, by tin and solders, with resulting weakening effect, occur at stresses insufficient to produce a large plastic deformation. In all cases tin has a greater effect than have the solders, and of two solders, that with the higher tin content has the greater effect. The fractures were accompanied by the formation of cracks which were usually intercrystalline. Possible explanations are discussed.

W. HUME-ROTHERY.

Recrystallisation of silver, copper, and aluminium. R. GLOCKER and H. WIDMANN (Z. Metallk., 1927, **19**, 41—43; cf. Glocker and Kaupp, B., 1924, 1016; 1926, 15; and Glocker, A., 1925, ii, 272).—On annealing severely cold-worked metals, recrystallisation usually takes place in two stages, in the first of which the new crystals are formed irregularly throughout the mass, and in the second they become regularly oriented according to the laws of crystallography. In certain cases, however, one or the other of these stages does not occur; *e.g.*, commercial aluminium (98% Al) recrystallises directly in the second stage at 275°, whereas cold-worked copper that has been previously hot-rolled never recrystallises with regular orientation of the crystals. Intermediate annealings during rolling also appreciably affect the course of recrystallisation of some metals by lowering the temperature at which this action begins, and by suppressing one of the stages of recrystallisation. Small quantities of impurites have a similar action; *e.g.*, the presence of 0·05% Fe in silver lowers the recrystallisation temperature to 20° or less, so that the severely cold-worked metal undergoes recrystallisation after storing for varying periods and intercrystalline fractures occur; this action may be suppressed by the addition of 0·1% Cu, which raises the temperature of recrystallisation to about 150°. Larger amounts of copper raise this temperature still further; with 0·3% Cu it is over 220°.

A. R. POWELL.

Effects of impurities on copper. III. Effect of arsenic on copper. IV. Effect of arsenic plus oxygen on copper. D. HANSON and C. B. MARRYAT (Inst. Metals, March, 1927, advance copy, 43 pp.).—The effect on copper of up to 1% As is investigated in the absence of other impurities. Sound castings of these alloys are difficult to obtain. The alloys are remarkably malleable and ductile, and can be worked hot or cold to any desired extent. Within the range of composition investigated arsenic produces only a very slight increase in the hardness, tensile strength, and ductility of copper; the effect of cold-rolling increases slightly with the arsenic content. The fatigue range is relatively high; the ratio of fatigue range to ultimate stress is approximately 0·9, due, possibly, to the great ductility of the material and its capacity for undergoing cold-work. The electrical conductivity falls rapidly as the arsenic content increases. Arsenic reduces the crystal size in castings, but this effect is not appreciable in annealed and worked material. The solubility of arsenic in copper is about 7·25% at all temperatures. In the case of alloys containing up to 2% As and 0·1% O, no difficulty was experienced in obtaining sound castings. The addition of arsenic to copper containing oxygen counteracts the embrittling effect produced by the latter element, and makes cold-rolling possible. Ultimate stress measurements show that, if the ratio of arsenic to oxygen is less than 10 : 1, the process of cold-work cannot be carried very far without damaging the material; it becomes "over-rolled." This relation indicates quantitatively the beneficial effect of arsenic in correcting the loss of ductility of copper due to oxygen; it is suggested that an increase in the arsenic content in commercial arsenical copper might be permitted with advantage. Copper containing arsenic and oxygen can be rolled hot without difficulty. The addition of oxygen to copper containing arsenic makes little difference to the results of mechanical tests and electrical conductivity measurements. In alloys of low arsenic content the oxygen exists as cuprous oxide; as the arsenic content increases up to 1% the cuprous oxide becomes darker, and is gradually replaced by a slate-grey constituent, which is generally associated with cavities, and which is shown to be a reaction product between cuprous oxide and arsenic; when present in small quantities it is not harmful to copper. Arsenic raises the softening temperature of cold-rolled strip very considerably, irrespective of the oxygen content, a small amount having nearly as much effect as 1%. Annealing in hydrogen shows that copper containing more than a trace of oxygen becomes embrittled by reducing gases, irrespective of the arsenic content.

M. E. NOTTAGE.

Effects of impurities on copper. V. Effect of bismuth on copper. D. HANSON and G. W. FORD (Inst. Metals, March, 1927, advance copy, 10 pp.).—Copper–bismuth alloys containing 0·001—0·1% Bi were examined in which the oxygen content is kept as low as possible. Sound castings of these alloys were not easy to obtain owing to excessive oxidation; a process of bottom-pouring was finally used. The solid solubility of bismuth in copper was found to be less than 0·002%, the bismuth, when present in sufficient quantity, being found in the crystal boundaries. Small quantities of bismuth adversely affect the rolling properties of copper, particularly during hot-rolling, the limiting amount is less than 0·01%, whilst for cold-rolling it is about 0·05%. Tests on rolled bar showed that bismuth has a harmful effect when present in still smaller quantities, particularly on the cold-working properties; in material which has to withstand severe cold-work, bismuth should be excluded, whilst for most other purposes the maximum should be 0·005%. Bismuth lowers the electrical conductivity of copper slightly, but its effect is negligible within the limits that permit successful working. The effect on the ultimate stress and the fatigue range is also small. In the notched bar (Izod) tests, the energy absorbed at fracture appeared to be less than that for bismuth-free copper of similar oxygen content.

M. E. NOTTAGE.

Brittleness in arsenical copper. C. BLAZEY (Inst. Metals, March, 1927, advance copy, 15 pp.)—Examination of cases of failure which occurred spas-

modically in arsenical copper tubing showed that brittleness was only developed after heating below 600—650°. The tensile strength in the brittle condition is much below normal, the elongation relatively slight, and the fracture non-necked and intercrystalline; this latter observation suggests that the brittleness may be due to the temporary setting aside to the boundaries of a constituent which interferes with the rearrangement of the copper atoms at recrystallisation, but which can be redissolved after the rearrangement has taken place. The bend test readily disclosed the brittleness in thin strip and wires. In wire form, the metal behaved well under twist test. Brittle samples could be cold-rolled but not cold-drawn. Susceptibility to brittleness is inherent in the metal, and 500° appears to be the critical temperature for developing it; it disappears rapidly at 600°. In the tensile test the rates of elongation and hardening were practically identical for susceptible and non-susceptible metal, and for any one sample in the brittle and the ductile states, which suggests that the weakness resides in the grain boundaries, as, if the grains themselves were different, differences in the rates of extension or hardening would have been found. Continued cold-drawing when in the brittle condition brought about a progressive lowering of tensile strength and false increase in elongation, due to the opening up of numerous minute fissures. Rate of cooling from the annealing temperature, ageing at room temperature for 1 year, the influence of the furnace atmosphere (oxidising or reducing), chemical constitution, and microstructure had no apparent connexion with the susceptibility or non-susceptibility to brittleness. It appears probable that the susceptibility is connected in some way with the melting operations, because susceptible material can be converted into non-susceptible by re-melting and re-poling. M. E. Nottage.

Copper–beryllium alloys. M. G. Corson (Brass World, 1926, **22**, 314—320; Chem. Abstr., 1927, **21**, 47).—Beryllium may be used as a hardening agent for copper. The maximum solubility of beryllium is 1·7%. From 3·8 to 9·5% Be, the crystals first obtained belong to a new type of crystal lattice corresponding with the β-phase, having a body-centred cubic lattice. The β-phase is unstable in that the maximum copper content cannot be kept constant through all temperatures, and that beryllium shows extreme activity in the molten state. Ternary copper–nickel–bismuth alloys exhibit a smaller tendency for precipitation of a secondary constituent than do copper–nickel–silicon alloys, any such precipitation being sub-microscopical. The properties of a copper–nickel–beryllium alloy containing 0·3% Be in the wrought and heat-treated state are listed. A. A. Eldridge.

Sodium cyanide copper solutions. W. D. Bonner and B. D. Kaura (Chem. Met. Eng., 1927, **34**, 84—85).—A cupric salt in a copper plating electrolyte is wasteful since cyanide is used up to reduce it to the cuprous form, whilst of the cuprous salts available, the cyanide, although more soluble than the chloride in sodium cyanide, is so susceptible to atmospheric oxidation that much free cyanide must be present. An electrolyte containing cuprous chloride is the best, being more stable and yielding higher current efficiencies. The molecular ratio of cyanide to copper must not exceed 5, for when the amount of copper is less than 1 mol. to each 5 mol. of cyanide, solution of copper in the cyanide solution takes place and not deposition. Agitation of the bath by bubbling hydrogen, nitrogen, or coal gas freed from carbon dioxide raises the current efficiency, whereas mechanical or air agitation causes oxidation. W. G. Carey.

Etching reagent for copper. B. W. Holman (Inst. Metals, March, 1927, advance copy, 1 p.).—A small, dry crystal of silver nitrate is placed on the polished surface to be etched, and a drop of water allowed to fall on it. A light deposit of silver is formed and turns grey in about 3 sec., when both deposit and crystal are washed off with a jet of water. A clean, evenly-etched surface is obtained free from silver, which tends to adhere if silver nitrate solution is used. A very fine structure is shown under high magnification. W. Hume-Rothery.

Crystallisation of the lead–tin eutectic. F. Hargreaves (Inst. Metals, March, 1927, advance copy, 2 pp.).—Straining and etching methods applied to sections cut from a 30 lb. ingot of lead–tin alloy of eutectic composition showed that the outside was composed of large chill crystals about 0·15 in. long, which showed a laminated structure and no tendency to "colony" formation. Crystals of similar size but with a coarser structure were found adjacent to the chill crystals, and the middle of the ingot was composed of small crystals up to 0·05 in. in diameter, in which the "colony" type of structure was well developed. The transition from large to small crystals was sudden, but the coarsening of the eutectic structure was gradual. Large nodules possessing a coarse radial structure comprised the upper layer. M. Cook.

Silver contents of Roman lead from Folkestone and Richboro Castle. J. N. Friend and W. E. Thorneycroft (Inst. Metals, March, 1927, advance copy, 2 pp.).—A specimen of Roman lead from Folkestone contained 0·0072% Ag, with traces of bismuth, and possibly also copper and antimony. A specimen from Richboro Castle contained 0·0078% Ag, with slightly more bismuth than in the Folkestone specimen, traces of copper, antimony, and tin being indicated. W. Hume-Rothery.

Influence of calcium on aluminium containing silicon. J. D. Grogan (Inst. Metals, March, 1927, advance copy, 13 pp.).—Aluminium alloys were prepared containing calcium and silicon in the ratio 1 : 1·4 as demanded by the compound $CaSi_2$; a similar series was prepared containing silicon alone, also two alloys containing, respectively, 1 and 2% Ca without any added silicon. Calcium forms with aluminium the compound $CaAl_3$ as a network round the primary aluminium grains and containing 8·5% Ca. When silicon is added to calcium–aluminium alloys, a new constituent, thought to be $CaSi_2$, appears, possibly containing aluminium either in combination or in solid solution, and resembling $MgSi_2$ in colour. Its solubility in solid aluminium is extremely low, but unlike the latter compound, $CaSi_2$ does not impart the property of age-hardening to the alloy. It is formed in preference to $MgSi_2$ in alloys containing calcium, silicon, and magnesium; conse-

quently, calcium is able to suppress the age-hardening phenomena associated with $MgSi_2$ unless excess of silicon be present. Microscopical examination shows that in alloys containing calcium and silicon in the ratio required by the compound $CaSi_2$, this constituent appears; addition of calcium to such alloys causes the appearance of $CaAl_3$ in addition to $CaSi_2$; no additional constituents of the types CaSi and Ca_2Si appear. Electrical conductivity measurements show that the limit of solid solubility of silicon in aluminium at room temperature is about 0·6%, at which concentration the conductivity curve flattens out; the tensile strength of the annealed material also shows a similar change at this composition. The addition of calcium to aluminium containing silicon rapidly raises the electrical conductivity until calcium and silicon are in the proportions required by the compound $CaSi_2$; additional calcium causes the conductivity to fall slowly. The addition of silicon to aluminium containing calcium produces little change in the conductivity until sufficient has been added to unite with all the calcium to form $CaSi_2$; further calcium causes the conductivity to fall rapidly. M. E. NOTTAGE.

Determination of calcium in aluminium alloys. P. G. WARD (Inst. Metals, March, 1927, advance copy, 1 p.).—The alloy (2 g.) is dissolved in caustic soda, the solution diluted, and the precipitate after settling is filtered off, dissolved in hydrochloric acid, neutralised with ammonium solution, and the iron and aluminium are precipitated with excess of 10% ammonium benzoate solution. The calcium in the filtrate is precipitated as oxalate and weighed as oxide. M. E. NOTTAGE.

Solubility of silicon in aluminium. W. KÖSTER and F. MÜLLER (Z. Metallk., 1927, **19**, 52—57).—The ratio of graphitic to soluble silicon in silicon–aluminium alloys varies with the heat treatment to which the metal has been subjected, and this is taken to indicate differences in the solubility of silicon in aluminium with the temperature. From chemical analyses, conductivity tests, and micrographical examination it is concluded that the solubility of silicon in aluminium rises linearly with the temperature from less than 0·1% at 300° to a maximum of 1·7% at 570°. The dissolved silicon is converted to silica on treating the metal with acid, whereas that present as eutectic or as inclusions of graphitic silicon remains as a black or brown powder. The greater the proportion of dissolved silicon in commercial aluminium the greater is its resistance to corrosion. A. R. POWELL.

Transformation of zinc–aluminium alloys in the solid state. W. FRAENKEL and J. SPANNER (Z. Metallk., 1927, **19**, 58—60).—Aluminium–zinc alloys of the composition Al_2Zn_3 were annealed for 8 days at 380° and quenched, the time taken for the maximum rise of temperature associated with the spontaneous decomposition of the compound being measured. Addition of 0·5% Bi, Pb, Sb, or Ag had no effect on the action, whereas small proportions of copper, tin, or cadmium retarded the rate of decomposition without appreciably affecting the maximum temperature reached. Lithium and magnesium rendered the action so slow that no rise in temperature took place. In every case the retardation was greater the greater the amount of impurity present, and this fact probably explains the conflicting results of other investigators of the α and γ ranges in the zinc–aluminium system. A. R. POWELL.

Reports of investigations: ore dressing and metallurgical laboratory. C. S. PARSONS, J. S. GODARD, and R. K. CARNOCHAN (Canad. Dept. Mines, 1925, [670], 11—71).—The article contains seventeen reports dealing with experimental work on the concentration by flotation, table treatment, etc. of a number of lead, copper, zinc, gold, and silver ores, and on a pyritic molybdenite ore from various Canadian mines, and includes data on selective flotation of zinc–lead–copper ores. Thiocarbanilide in a pulp made alkaline with sodium carbonate usually gave a good lead concentrate, and zinc could subsequently be floated by addition of copper sulphate and potassium xanthate; the presence of cyanide reduces the zinc content of the lead concentrate much more than does sodium sulphite or phosphate. A. R. POWELL.

Reports of investigations: electrochemical and hydro-metallurgical laboratories. Hydro-metallurgical treatment of iron sulphide ores for the production of electrolytic iron and of pyrrhotite of low gold and copper content. Treatment of ilmenite for the recovery of electrolytic iron and titanium oxide. R. J. TRAILL and W. R. MCCLELLAND (Canad. Dept. Mines, 1925, [670], 72—88).—Further tests on the wet treatment of pyritic ores for the recovery of electrolytic iron and elemental sulphur are recorded (cf. B., 1926, 670). The ferrous chloride solution obtained after leaching with ferric chloride the product obtained by heating the ore at 750° in a neutral atmosphere may be purified from most heavy metals by heating with powdered ferrous sulphide, but zinc cannot be precipitated in this way, and is co-deposited with the iron in the subsequent electrolysis. For this operation the electrolyte should have an acidity of p_H 3·7—3·9 and a temperature of 80—90°; a carbon anode and a rotating steel cathode are used, separated by an asbestos cloth diaphragm. From electrolytes containing only traces of zinc a deposit containing 99·94% Fe has been obtained with an efficiency of 93%. The method has been applied to an ore containing 1·2% Cu together with pyrrhotite, pyrites, and gold; heating at 750° in a neutral atmosphere caused some alteration to the pyrrhotite which resulted in a poor extraction. Better results were obtained by leaching out the pyrrhotite directly and heating the residue at 750° preparatory to a second leaching. Nearly 90% of the copper and iron were recovered together with 60% of the sulphur; cyanidation of the residue gave a gold extraction of over 75%. The process has been adapted to the extraction of titanium dioxide and iron from ilmenite; the ore is heated with carbon at 950° to obtain a sponge of metallic iron containing the titania in mechanical admixture. The product is separated from gangue material by magnetic treatment, and the magnetic product is leached with ferric chloride solution to obtain ferrous chloride for electrolysis after a preliminary purification with calcium sulphide. The insoluble residue from leaching is utilised for the recovery of titania. A. R. POWELL.

Combined pyro- and hydro-metallurgical process

for the treatment of nickeliferous pyrrhotite and other sulphide ores for the recovery of iron and sulphur in addition to the other contained metals. H. C. Mabee and A. E. Smaill (Canad. Dept. Mines, 1925, [670], 89—94).—The ore is smelted with an alkali flux under non-oxidising conditions to obtain a low-grade matte containing all the copper, nickel, and precious metals. This matte, on exposure to the air, disintegrates into a fine powder, which is roasted in a two-stage mechanically-rabbled furnace, in the first stage of which the temperature is maintained at 400° and in the second stage at 600°. The product is mixed with 10% of its weight of sodium chloride, damped with water, and again roasted at 400°, the issuing vapours being absorbed in water. This water is then used to leach the roasted product, and leaching is continued with 10% sulphuric acid. The residue contains practically all the iron and the greater part of the nickel, but only a small proportion of copper and sulphur; it is amenable to direct smelting for the production of nickel steel. The leach liquor is electrolysed for the recovery of copper, then treated for the removal of iron, and again electrolysed to recover the nickel. Over 90% of the copper and 40% of the nickel are obtained as pure metals. A. R. Powell.

XV.—LEATHER; GLUE.

Official methods for analysis of vegetable-tanned leather (J. Soc. Leather Trades' Chem., 1926, **10**, 411—418).—The number of individual pieces to be sampled shall be $n = 0{\cdot}7\sqrt{X}$ where X = total number of pieces, not be less than 3 (methods of taking the sample are given). *Moisture.*—Dry 2 g. of the well-mixed pieces to constant weight at 100—103°. Greasy leathers must be degreased before drying. *Total ash.*—Incinerate 5 g. of leather in a tared dish at a dull red heat in a muffle furnace until all carbon is consumed. *Ash of solubles.*—Ignite the residue from total soluble matter in water-soluble matters determination (*q.v.*). *Oils and fats.*—Extract 20—25 g. of leather in a Soxhlet apparatus with light petroleum (b.p. 40—65°) until free from grease, and dry fatty matter at 95—100° to constant weight. Leathers which contain oils insoluble in light petroleum must be extracted a second time with anhydrous ether. *Water-soluble matter.*—(*a*) After the fat extraction, the leather is completely freed from solvent by leaving it exposed to the air (not heated), transferred to a Procter extractor, covered with distilled water, left overnight, and extracted at 45° by continuous extraction until 1 litre of extract has been obtained in 3—3½ hrs. The total solubles and non-tans are determined in the extractive by the usual official method. (*b*) The fat-extracted leather, freed from solvent, is divided into two equal parts, each shaken for 5 hrs. with 500 c.c. of distilled water at 18°, and the liquid is filtered and analysed as before. *Nitrogen and hide substance.*—Heat 1·5 g. of the leather pieces with 15—25 c.c. of strong sulphuric acid for 5—15 min., add 10 g. of powdered potassium sulphate and 0·5 g. of anhydrous copper sulphate, boil for 1 hr. after the liquid has become clear and nearly colourless, dilute with about 200 c.c. of water, add 50 c.c. of 35% caustic soda solution, distil off the ammonia into standard acid, and titrate with standard alkali, using carminic acid, alizarin red S, or methyl red as indicator. *Glucose.*—Mix 200 c.c. of leather extract with 25 c.c. of a saturated solution of lead acetate, shake frequently during 5—10 min., filter, add excess of solid potassium oxalate to the filtrate, mix, and filter, returning filtrate until clear. To 150 c.c. of this filtrate add 5 c.c. of strong hydrochloric acid and boil under reflux for 2 hrs., cool, neutralise with anhydrous sodium carbonate, make up to 200 c.c., and filter through a double filter. Determine the dextrose immediately by adding 50 c.c. of the clear neutralised solution to 25 c.c. each of the ordinary Fehling's solutions. Heat to boiling in exactly 4 min., boil for 2 min., filter immediately through asbestos, wash thoroughly with hot water, then alcohol, finally ether, dry for ½ hr. in water oven, and weigh as cuprous oxide. *Epsom salts.*—Ash 5—10 g. of leather, moisten with water, add 15 c.c. of strong hydrochloric acid, wash and dilute to 50—75 c.c., add 2—3 drops of nitric acid, boil, nearly neutralise with 1 : 1 ammonia solution, then add slight excess of weak ammonia solution. Boil for a few minutes, filter, wash the precipitate with hot water. Add 10 c.c. of a saturated solution of ammonium oxalate to the boiling filtrate, cover, allow to settle for 2 hrs. in a warm place, allow to cool, and make up to 250 c.c. An aliquot portion (equivalent to 2 g. of original leather) is made up to 150 c.c., acidified with hydrochloric acid, a slight excess of clear saturated sodium ammonium hydrogen phosphate solution is added, and the mixture rendered faintly ammoniacal. After 15 min. add 5 c.c. of strong ammonia, and keep overnight. Determine magnesium gravimetrically as $Mg_2P_2O_7$. *Mineral acidity.*—Weigh 2 g. of the leather in a platinum or rhotanium dish, add 40 c.c. of $0{\cdot}1N$-sodium carbonate solution, mix thoroughly, evaporate to complete dryness on steam bath, ignite at dull red heat, moisten with 25 c.c. of hot water, break up the residue, filter, and wash 4—5 times with hot water. Dry, and ignite filter paper and residue at dull red heat, cool, and add to the residue 40 c.c. of $0{\cdot}1N$-sulphuric acid, cover the dish, place on steam bath for 30 min., filter, if necessary, into the first filtrate, washing the paper free from acid with hot water. Cool the solution and titrate with $0{\cdot}1N$-alkali and methyl orange.

D. Woodroffe.

Urunday and urunday [tannin] extract. W. Vogel (Collegium, 1926, 535—541).—Urunday or urunday pardo (*Astronium Balansæ*, Engl.), belonging to the *Anacardiaceæ* family, is very similar to quebracho colorado chaqueno. Urunday tannin belongs to the pyrocatechol group, and gives the same qualitative reactions as quebracho. Commercial urunday extracts show a bluish-violet fluorescence compared with the yellowish-green of quebracho solutions. The natural products gave no difference either in acid or alkaline solution, and the above bluish-violet fluorescence is not considered a characteristic of urunday. The composition of the heartwood, sapwood, and bark are: tans, 14·4, 1·4, 12·4%; non-tans, 1·5, 1·2, 3·3%; water, 19·0, 42·5, 12·4%. Urunday extract is manufactured in the same way as quebracho extract; 100 lb. of urunday wood yield 20 lb. of solid extract containing tans 63—65%, non-tans 6·1—7·2%, insolubles 5—8%, and ash 1·6—1·9%. The tans/non-tans ratio is slightly lower than

that for quebracho, 100 : 10 against 100 : 7. A sulphited extract is prepared with composition :—tans 67·2%, non-tans 10·5%, insolubles 0·3%, and ash 7·5%. Sulphited urunday extract always contains some insoluble matter, thus distinguishing it from sulphited quebracho. There is very little difference between leathers tanned with urunday and quebracho, respectively, until they have been exposed to light, when the urunday-tanned leather becomes a pure brown. The colour of leather tanned with sulphited urunday extract does not differ from that of leather tanned with the natural extract, but it darkens more on exposure to light. Urunday excels quebracho in giving greater firmness and better weight. They are of equal penetrating power.

D. WOODROFFE.

Biological hydrolytic units from protein complexes. E. KANN (Collegium, 1926, 541—545).—Bromopropionylalanine was treated in the cold with a dehydrating agent. A cyclic compound separated out, which lost its bromine, and, on treatment with dilute hydrochloric acid, gave up its nitrogen and yielded racemic acid. This is the product obtained by the action of enzymes on alanine. Bromopropionylaspartic acid behaves similarly, also the halogenoacyl derivatives of tyrosine, phenylalanine, and arginine.

D. WOODROFFE.

Standardised bacterial preparations in the leather industry. E. LENK (Collegium, 1926, 556—560).—Bating materials should be tested on three kinds of protein at least, viz., casein, collagen or gelatin, and elastin. Gelatin is a favourable substrate. Samples of "Cutrilin" (a bacterial bate) and pure pancreatic bates were kept at 35° for several days, and the content of trypsin was determined at intervals. The pancreatic bates lost 60% strength in 1 day, whereas the "Cutrilin" was 50% strong after 7 days. The action of the trypsin in "Cutrilin" was not diminished so much by change in p_H value as that in pancreatic bates. "Cutrilin" showed maximum effect from p_H 5—9, and decomposed gelatin substrate more quickly than pancreatic bates at p_H values less than 7·3.

D. WOODROFFE.

A tanning accelerator. R. E. LIESEGANG (Collegium, 1926, 571).—Alkaline pyrogallol or pyrocatechol solutions render insoluble those portions of the silver bromide–gelatin layer which have been exposed to light, and therefore can be reduced. Large amounts of sodium sulphite, which are usually present in developers, restrain this tannage. The author placed a granule of silver chloride (diam. 5 mm.) in a 10% gelatin solution, and covered the gelatin with a concentrated "metol-adurol" developer which was renewed every 4 days for 2 months. Then the gelatin was melted to remove the granule, but a layer of insoluble gelatin 3 mm. thick surrounded the latter. A portion of the molten jelly was mixed with some silver chloride paste, allowed to set, and in $\frac{1}{2}$ hr. it was insoluble in water, whilst the untreated portion was soluble. The silver chloride catalysed the tanning effect in spite of the sulphite in the developer.

D. WOODROFFE.

Theory of oil tannage. B. N. MATHUR (J. Amer. Leather Chem. Assoc., 1927, **22**, 2—44).—It has been shown that of two samples of seal oil, a pale coloured and a very dark brown, the latter possessed superior tanning properties and yielded much stronger leathers. Tanning tests with the fatty acids of seal oil showed that acrolein was not necessary in chamoising, but that the unsaturated fatty acids had tanning properties. Experiments on partially-tanned chamois leather showed that the presence of water is necessary to obtain the best leather. Hide substance, in order to be converted into a strong oil leather, requires a certain amount of moisture during the tanning process. Pieces of cotton cloth treated with the unsaturated fatty acids of seal oil and exposed to heat remained greasy unless they were kept moist. The carboxyl-groups of the unsaturated fatty acids do not undergo any chemical change during the process of tanning, and the hydroxyl-groups produced during the oxidation unite with the hydrogen of the hide substance to form water. The soaps of unsaturated oils do not change into lactones under the conditions of chamoising. Sodium salts of the unsaturated fatty acids of seal, cod-liver, and shark-liver oils, in the presence of air and moisture form compounds quite different from those formed in an atmosphere of dry air alone. The brownish compounds developed in air under the influence of moisture on the sodium salts of the above fatty acids are capable of tanning and essential to the chamoising process. Air is not absorbed during oil tanning. The hydroxy-compounds are formed when the seal oil contains about 30% of moisture, and they combine with the pelt without the evolution or absorption of gas and form chamois leather. Any unsaturated fatty acid will tan, provided it can combine with a hydroxy-group from the moisture. The sodium salt of dihydroxystearic acid has tanning properties; α- and β-hydroxystearic acids do not tan. The best tanning agents among the fatty acids of the oleic acid series are those in which the unsaturated linkage is in a reactive place, *e.g.*, positions 10 and upwards. These will combine with water even at ordinary temperatures. Hence the superiority of fish oils in tanning. The unsaturated oils capable of chamoising first hydrolyse on the hide fibre, and the rise in temperature facilitates the hydrolysis. The unsaturated acids thus set free, having at least one double linkage in a reactive position, combine chemically with the hydroxyl of the moisture. The saturated acids thus formed, chiefly monohydroxy-acids, combine at 50° with the hide substance with the elimination of water at a fairly rapid rate. The hydroxy-acids containing two or more hydroxyl-groups in the molecule combine with two or more mols. of hide substance and produce leather of inferior quality and feel. In oils used for tanning purposes, acids containing a single double-bond are those most essential in chamoising.

D. WOODROFFE.

Nature of one-bath chrome tannage. K. H. GUSTAVSON (Ind. Eng. Chem., 1927, **19**, 81—83).—Hide powder tanned with a basic chromium sulphate solution (66% acidity and 2·1 g. Cr_2O_3/litre) absorbed 3·82% of chromium trioxide, and when this tanned powder was treated with an oxalato-chromium compound it absorbed a further 4·66%; untreated hide powder absorbed 5·15% from the latter liquor. Hide powder, which absorbed 5·46% of chromium trioxide from an oxalato-chromium solution and 7·09% from a basic

chromium sulphate solution (5 g. Cr_2O_3/litre), was tanned with the latter, and then treated with the oxalato-chromium solution and a further lot of stronger basic chromium sulphate, respectively. It absorbed a total of 11·36% of chromium trioxide instead of 12·55% with the basic chromium sulphate followed by the oxalato-chromium compound. It absorbed a total of 10·25% of chromium trioxide instead of 17·47% when treated with a weak basic chromium sulphate followed by a stronger one. Hide powder tanned with a basic chromium sulphate (63% acidity and 11·8 g. Cr_2O_3/litre) absorbed 17·43% of chromium trioxide, and when afterwards treated with the oxalato-chromium solution, the absorbed chromium increased to 20·96% instead of 22·58% as calculated. The stronger solution contained some anodic chromium complexes. Hide powder pretanned with the oxalato-chromium and then with a basic chromium sulphate absorbed 10·37% Cr_2O_3 instead of 15·41% as calculated. Apparently tanning with cationic chromium compounds does not involve the same protein groups as anionic tannages. Anionic chrome-tanned powder has not the same affinity for cationic chromium compounds as untreated hide powder. Such reactions affecting the basic protein groups appear to inhibit the fixation of cationic chromium. The fixation of basic chromium sulphate by hide protein comprises two separate, but mutually influenced processes, the combination of cationic chromium by means of the acidic protein groups, and of the hydrolysed acid by the basic protein groups. Chemical inactivation of the latter reduces the acid-fixative capacity of the protein, consequently the chromium fixation is decreased. It is possible that the chemical interaction with the basic protein groups induces structural rearrangements and redistribution of the valency forces. The data given support the view of the dual nature of anionic and cationic chromium fixation. Cationic chromium combines with the acidic groups of the hide protein, forming a very stable salt. Anionic chromium forms molecular compounds with the basic protein groups.

D. WOODROFFE.

Behaviour of formaldehyde-tanned hide powder toward chromium compounds. K. H. GUSTAVSON (Ind. Eng. Chem., 1927, **19**, 243—248).—Portions of hide powder tanned in formaldehyde solutions of different concentrations respectively at p_H 8·0 and then treated with chromium sulphate solutions, absorbed less chromium as the concentration of the formaldehyde solution increased. The diminution in chromium fixation was more marked with a chrome liquor containing cathodic and anodic chromium than with cathodic chromium alone. Formaldehyde-tanned hide powder prepared at p_H 7·0—8·0 fixed less chromium than powders tanned at other p_H values. Formaldehyde-tanned hide powder tanned at p_H = 7·0 absorbed less alumina from a basic aluminium sulphate solution and less tannin from a solution of hemlock bark extract. The anionic-sulphito and oxalato-chromium compounds were less fixed by formaldehyde-treated hide powder at p_H 7 and 8 than by untreated hide powder. The fixation of these complexes is partly by the basic protein groups. The formaldehyde treatment reduces the number of basic groups. The tanning mechanism of basic chromium sulphates consists of two separate but mutually influenced reactions, the fixation of the complex cations by the acidic protein groups and the fixation of the hydrolysed acid by basic protein groups. The diminution of the number of basic protein groups by the formaldehyde treatment at p_H = 7—8 diminishes the fixation of hydrolysed acid and thus represses the hydrolysis of the chromium salt, hence less chromium is fixed. Increased chromium fixation was observed with hide powder tanned with formaldehyde at p_H 12·2. This is attributed to the formation of alkali collagenate which leads to an increased activity of the acidic groups in the hide protein. There is also a considerable deaggregation of the protein. Chromium sulphates of high acidity and low concentrations gave practically the same fixation by hide powder, untreated and treated with formaldehyde at p_H values 7·0 and 12·2. With decreased acidity the differences in the chromium fixation became more pronounced. The more basic chrome liquors contain large quantities of anionic complexes which probably react with the basic protein groups. Cationic chromium fixation is less influenced because the reduced basic protein groups affect it indirectly only. Pre-treatment of the hide powder with formaldehyde at low p_H values, *e.g.*, p_H 2·5, is chiefly a process of peptisation and activation of basic protein groups. Hence, the more acid chromium salts are not affected by such pre-treatment, but the colloidal liquors give increased chromium fixation. The acidity of the resulting chromium–formaldehyde–collagen compounds was lower in all cases than the corresponding chrome–collagen compound. The experiments show the importance of the previous treatment of the collagen on its subsequent behaviour with tanning agents.

D. WOODROFFE.

Extraction of nitrogenous matter from calfskins by salt water. H. B. MERRILL (Ind. Eng. Chem., 1927, **19**, 249—251).—Samples of freshly-flayed and fleshed calfskin and skin cured with 25% of its own weight of salt and dried, were soaked in water, 0·125*N*, 0·25*N*, 0·5*N*, and *N*-sodium chloride, respectively, for 24 hrs., and then with a fresh portion of the same solution respectively for a further 24 hrs. The used solutions were analysed for nitrogen, and it was found that fresh calfskin contains a small amount of nitrogenous matter insoluble in water, but soluble in dilute sodium chloride. The quantity of material dissolved either by water or salt approaches a limit with increasing time or increasing salt concentration, indicating that collagen is only slightly attacked. After curing with salt, the amount of water-soluble matter is doubled, whilst that of salt-soluble matter is diminished slightly. With cured skin, the addition of salt to the solution has almost no effect on the amount of nitrogenous matter extracted.

D. WOODROFFE.

Influence of hydrogen-ion concentration and valency of added anion on plumping in tan liquors. R. O. PAGE and J. A. GILMAN (Ind. Eng. Chem., 1927, **19**, 251—252).—Pieces of soaked, limed, fleshed, and unhaired cow-hide butt were shaken with distilled water for 1 hr., the thickness was measured with a sensitive gauge, the pieces were shaken with water to restore their normal shape, and then put into a 12·5% solution of wattle-bark extract, to which had been added different

quantities of sulphuric, hydrochloric, or lactic acid, respectively. The thickness was measured again after 24 hrs., and the p_H value of the liquor determined electrometrically. There was a slight increase in plumping from the isoelectric point up to p_H 3·5, then a rapid increase to a maximum at p_H 1·8 or less, followed by a steady decrease in swelling with further reduction in the p_H. The plumping and p_H values were plotted on a graph. Lactic and hydrochloric acids give approximately the same curves, but the sulphuric acid curve was only about half as high as that for the others, showing that the plumping for sulphuric acid, which gives a bivalent anion, was half that for acids with a univalent anion. D. WOODROFFE.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

PATENTS.

Production of dicyclic bases. J. D. RIEDEL A.-G. (G.P. 433,098, 5.7.24. Addn. to 423,027; B., 1926, 513). —Secondary aliphatic bases containing the phenylethyl residue or derivatives of these compounds are condensed with α-halogeno-carboxylic acids or their esters, and the acids thus formed are converted into the acid halides, which, on treatment with condensing agents such as aluminium chloride, lose carbon monoxide and hydrochloric acid giving *N*-substituted tetrahydro*iso*quinolines. *E.g.*, from *N*-methylphenylethylamine and ethyl bromoacetate is obtained *ethyl* N-*methylphenylethylaminoacetate*, $CH_2Ph \cdot CH_2 \cdot NMe \cdot CH_2 \cdot CO_2Et$, b.p. 152—154°/12 mm., which, on hydrolysis with hydrochloric acid and treatment of the resulting hydrochloride with potassium hydroxide, gives N-*methylphenylethylglycine*, m.p. 163°. By the action of acetyl chloride and phosphorus pentachloride this is converted into the *glycyl chloride*, m.p. 98—100°, and the latter on treatment with aluminium chloride in the presence of benzene and warming for a short time at 100° gives the hydrochloride of N-*methyltetrahydro*isoquinoline, m.p. 161°. E. H. SHARPLES.

Separation of amine mixtures of partly hydrogenated aromatic compounds. I. G. FARBENIND. A.-G., Assees. of F. MAYER and K. SCHIRMACHER (G.P. 434,403, 6.3.25).—The mixtures are treated with formaldehyde in acid solution whereby the bases unsubstituted in the *p*-position are converted into diarylmethane derivatives. *E.g.*, the mixture of aminohydrindenes obtained by the nitration and reduction of hydrindene is heated in hydrochloric acid solution with 40% formaldehyde for about 3 hrs. at 70—90°. It is then made alkaline and distilled in steam; pure 5-aminohydrindene, m.p. 34—35°, distils, and the residue, a diaminodihydrindenylmethane, may be purified by crystallisation. Similarly, by the action of 40% formaldehyde on a mixture of 5- and 6-*ar*-tetrahydronaphthylamine, pure 6-*ar*-tetrahydronaphthylamine, m.p. 148°, and a diarylmethane derivative are obtained.
E. H. SHARPLES.

Preparation from carboxylic acids of the pyridine and quinoline series of derivatives which are easily soluble in water. CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING), Assees. of T. BRUGSCH and H. HORSTERS (G.P. 433,646, 15.9.23).—The acids are treated with alkali cholates. *E.g.*, 2-phenylquinoline-4-carboxylic acid is added gradually with shaking to a 0·1*N*-solution of sodium cholate at 50°. Up to 3·5 pts. of the acid can be dissolved without separation on cooling. The substances are used as medicaments. E. H. SHARPLES.

Preparation of α-indanone. COMP. NAT. DE MAT. COLOR. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIS ÉTABL. KUHLMANN, C. COURTOT, and J. KROLIKOVSKI (F.P. 608,101, 19.12.25).—Pure indene or the coal-tar fraction containing it is treated with hydrochloric acid, the impurities are removed by distillation *in vacuo*, and the residual α-chlorohydrindene is oxidised with sodium dichromate and dilute sulphuric acid to α-indanone, b.p. 125°/15 mm., m.p. 40—41°, in 70—85% yield.
E. H. SHARPLES.

Production of derivatives of oxindole-3-acetic acid and its homologues containing halogens substituted in the aromatic nucleus. CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING), Assees. of W. SCHOELLER and K. SCHMIDT (G.P. 433,099, 11.3.25).—The acids are halogenated by the customary methods. *Monoiodo-oxindole-3-acetic acid*, m.p. 204°, *di-iodo-oxindole-3-acetic acid*, m.p., 214°, *dibromo-oxindole-3-acetic acid*, m.p. 275°, *dichloro-oxindole-3-acetic acid*, m.p. 256°, *oxindoleacrylic acid*, m.p. 212° (from oxindolealdehyde and malonic acid), *oxindole-3-propionic acid*, m.p. 208° (by reduction of the preceding), and *monoiodo-oxindole-3-propionic acid*, m.p. 224°, are described. E. H. SHARPLES.

Preparation of water-soluble derivatives by means of salt-forming groups of substituted arylarsinic and arylstibinic acids and their corresponding oxides. I. G. FARBENIND. A.-G., Assees. of K. STREITWOLF and A. FEHRLE (G.P. 433,105, 24.6.24. Addn. to 413,147; B., 1925, 783).—Amino-, or aminohydroxyaryl-arsinic or -stibinic acids or their corresponding oxides are treated with aldoses or ketoses under conditions described in the main patent. Examples are, the condensation products of dextrose with 3-amino-4-hydroxyphenyl-1-arsinoxide and 3-amino-4-hydroxyphenyl-1-arsinic acid, galactose with *m*-aminophenylstibinic acid, lactose with *m*-aminophenylstibinoxide, and arabinose with *p*-aminophenylarsinic acid. Condensation may be suitably effected by heating the components in aqueous solution at 50—55° with the addition, if necessary, of a little sodium hydroxide.
E. H. SHARPLES.

Preparation of cadmium salts of phenols and phenol derivatives. CHEM. FABR. VON HEYDEN A.-G., Assees. of R. GEBAUER (G.P. 433,102, 25.11.24).—Cadmium salts of phenols and phenol derivatives are prepared by the usual methods for the preparation of metal phenoxides. The products possess a similar physiological action to that of the analogous bismuth compounds and may be used as dusting powders, and also in the form of oil emulsions for injection purposes. *E.g.*, aqueous solutions of cadmium sulphate and sodium phenoxide are mixed and the precipitate is washed first with water, then with 96% alcohol and dried at 50°. The product is a white, odourless, talc-like powder having the composition, $PhO \cdot CdOH$, and is suitable for the preparation of antiseptic powders. Cadmium *salts* of tribromophenol, salicylic acid, sulphosalicylic acid, guaiacol, guaiacolcarboxylic acid, and resorcinol are described.
E. H. SHARPLES.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 29, 1927.

I.—GENERAL; PLANT; MACHINERY.

PATENTS.

Method and apparatus for heating, cooling, and controlling reactions at high temperatures. C. FIELD, Assr. to CHEMICAL MACHINERY CORP. (U.S.P. 1,619,660—3, 1.3.27. Appl., [A] 4.3.20. Renewed 1.2.27; [B] 17.4.22; [C] 20.4.22; [D] 15.7.22).—The material under treatment is heated by the condensation of mercury vapour, and the temperature is regulated by the pressure of the vapour. That portion of the mercury condensed by doing useful heating flows to a reservoir or hot well, and thence to the mercury boiler. The other portion of the vapour passes through a separate condenser and flows back direct to the boiler. The pressure of the vapour may be regulated by a venting valve controlled by the temperature but not by the pressure, with provision of a second relief valve opening under pressure but unaffected by the temperature.

B. M. VENABLES.

Process and apparatus for reactivating adsorption media. METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 242,986, 10.11.25. Conv., 12.11.24).—The adsorption medium is regenerated by hot gases which are blown into it from a number of chambers buried in it, and the gases plus the adsorbed substance are collected in and drawn off through a similar number of chambers so situated that the substance to be removed has no chance of collecting in cooler zones of the absorbent. The adsorbent may be kept in motion while being regenerated, and the heat may be supplied otherwise than by heating the scavenging gases. B. M. VENABLES.

Rotary furnaces. A. LACORE and J. PIRON (E.P. 259,238, 1.10.26. Conv., 2.10.25).—A rotary tubular furnace intended for burning lime, cement, minerals, etc. is provided with two portions enlarged to about double diameter, situated between three portions of normal diameter. The lowest (small) portion comprises the clinkering zone, the next large one the calcining zone, and the upper large one a dehydrating zone.

B. M. VENABLES.

Rotary hearth furnaces. INTERNAT. GENERAL ELECTRIC Co., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (E.P. 259,570, 5.10.26. Conv., 6.10.25).—A continuously operating furnace is provided with one or more rotating annular hearths, and is heated electrically by resistors in the walls. Regeneration of heat is effected by radiation from the hot outgoing to the cold ingoing goods. If two hearths are used rotating in opposite directions, a single opening may serve for charging and discharging both hearths. If one hearth only is provided, there are two openings, and only one set of goods passes through the electrically-heated zone, but on the rest of the circle between the two openings two sets of goods, outcoming and ingoing, are present on the hearth together.

B. M. VENABLES.

Apparatus for drying and heating. E. C. R. MARKS. From GRASSELLI CHEMICAL Co. (E.P. 265,771, 9.2.26).—A furnace similar in construction to a Herreshoff roaster of the muffled type, *i.e.*, the main heating gases do not make contact with the material, is arranged so that the air or gas which is used to cool the rabbles is withdrawn by a fan and supplied to the upper or drying hearth in contact with the material and under slight positive pressure. B. M. VENABLES.

Means of pulverising and separating all kinds of cereals, minerals, and the like. J. C. CARLINE (E.P. 265,841, 17.6.26. Addn. to E.P. 244,146; B., 1926, 112).—A modification of the grinding and screening apparatus described in the original patent, mainly consisting in that the revolving brush rubs against a wire screen. B. M. VENABLES.

Reduction of finely-divided material. W. GARDNER (U.S.P. 1,619,295, 1.3.27. Appl., 14.4.26).—The material is removed from a grinding mill before it has all been reduced to the finished size by an air stream of low velocity. The oversize is removed from the air stream in a settler and returned to the mill, and the finest material is collected in an air filter, the air not being returned to the grinding unit. B. M. VENABLES.

Centrifugal treatment of liquids. K. J. SVENSSON and K. A. P. NORLING (E.P. 264,130, 3.9.26. Conv., 11.1.26).—A centrifugal separator provided with feed pump and electrical heater is arranged to have a supply of "wash" liquor which, in addition to serving the ordinary purpose of entraining solid particles and removing them from the main liquid being treated, will serve also to prevent the electrical heater burning out should the supply of main liquid cease. Claim is made for various methods of connecting up the pump or pumps, reservoirs, and heaters. The wash liquor may be returned for re-use. B. M. VENABLES.

Filtration. [Separation of liquids.] H. S. HELE-SHAW and J. A. PICKARD (E.P. 265,674, 9.11.25).—A mixture of two liquids—preferably after separation as far as possible by other means—is passed through a filter comprising loosely packed solid threads of inorganic non-absorbent material (*e.g.*, asbestos, glass wool) to which the last trace of one liquid will adhere by surface tension even if it is very finely divided. The filter is cleaned by steam under pressure. B. M. VENABLES.

Filters. C. E. and W. FOX (E.P. 265,856, 16.7.26).—A telescopic tube is perforated and wound with a spiral

of wire with interposition of gauze or other filtering medium between the wire and the tube if desired. The whole is enclosed in a pressure-tight casing to which the prefilt is supplied. The filtrate is removed through a conduit passing from the interior of the tube, through a gland in the end of the casing, to the outside, and sediment may be dislodged from the filter medium by pulling this conduit, thus pulling apart the telescopic tube and simultaneously opening the spirals of wire.

B. M. VENABLES.

Drying liquid material. Apparatus for drying atomised material. OESTERREICHISCHE LANDWIRTSCHAFTS-GES. (Austr. P. 103,995—6, 21.9.21).—(A) The material under treatment is discharged, after previous atomisation, into the conduit conveying air to the drying chamber, through a tube entering it at an acute angle, so that the material is still more finely divided. (B) Air for drying the material enters through a filter at the bottom of a tower-shaped drying chamber, and the material is charged in through an opening at the lower end of the sloping partition at the top of the tower.

L. A. COLES.

Continuous separation of gas mixtures. H. WADE. From N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 266,396, 23.9.25).—The apparatus comprises a series of elements in each of which partial separation is effected by diffusion. The elements are so connected that the quantity of gas passing from one element to the next is approximately equal to that passing in the opposite direction. Pumps are provided between the elements to regulate the removal of the gases, and the resistance offered to the gases in their passage from one element to the other, caused by a porous wall in the one direction and by a number of tubes in the other, is regulated so as to be approximately the same in each case.

L. A. COLES.

Apparatus for the electrical purification of gases. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 435,093, 22.6.24).—The electrodes are constructed of corrugated sheet metal so arranged that the grooves are inclined to the horizontal and run obliquely to the direction of the gas stream. In treating chimney gases, the apparatus is placed in a conduit sloping upwards from the flue to the chimney. L. A. COLES.

Production of gas- and liquid-tight vessels and pipes. G. ADOLPH, A. PIETZSCH, and B. REDLICH (E.P. 252,366, 17.5.26. Conv., 22.5.25).—An acid- (or chemical-)proof container is constructed of sections of ceramic material, of single thickness and without external casing or support, the sections being joined only at their edges by means of an elastic binding medium composed of, *e.g.*, rubber or cellulose. The elastic medium may be used only on the inside portion of the joints and ordinary cement outside, or acid-proof cement inside and the elastic medium outside. B. M. VENABLES.

[Disappearing filament] optical pyrometer. C. E. FOSTER (E.P. 266,060, 19.11.25).

Detection of impurities in fluids (E.P. 266,047).—See XI.

Pyrometer (E.P. 266,060).—See XI.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Low-temperature carbonisation. Vertical retorts at H.M. Fuel Research Station. C. H. LANDER and J. F. SHAW (Dept. Sci. Ind. Res., Fuel Res. Tech. Paper No. 17, 7 pp.).—An account is given of the construction and performance of two settings of vertical cast-iron retorts designed for the low-temperature carbonisation of bituminous coal. Each se ting consists of two retorts, one pair being 21 ft. high and 6 ft. 6 in. × 4 in. in cross-section at the top, widening to 6 ft. 10 in. × 8 in. at the bottom; the retorts of the second setting are similar, but are 3 in. wider. The former pair proved to be too narrow for the continuous passage of small coal of caking quality; moreover, after being in operation a short time distortion occurred as a result of local overheating, and the retorts became unworkable. The wider retorts have been in satisfactory operation for 12 months at a working temperature of 625°, and with a throughput of 2½—4 tons per retort per day. The charge is not kept moving continuously, the extractor gear being operated for a short time at intervals of 1, 2, or 3 hrs., according to the nature of the coal being carbonised. The coke collects in a cooling chamber at the base of the retorts, and is discharged at intervals. The setting is heated by water-gas supplied to burners situated in corners built into the internal face of the setting so that the retorts are heated principally by radiation from the flames and brickwork, and direct contact of the flames with the walls of the retort is avoided. The consumption of water-gas is about 2500 cub. ft./hr. The tar yields vary from 11·1 gals./ton with Durham coal (run of mine) to 18·4 gals./ton with Garforth nuts. Excellent coke has been obtained from the fine coal (through a ⅜-in. screen), which amounted to 50—60% of the Durham coal used, by carbonising briquettes made with 74% of coal, 20% of low-temperature coke breeze, and 6% of pitch. The coke produced has proved very satisfactory as a domestic fuel. A. B. MANNING.

Caking power of coal. J. T. BURDEKIN (Dept. Sci. Ind. Res., Fuel Res. Survey Paper No. 8, 21 pp.).—"Caking-power curves" have been determined for a number of coals. 2 g. of powdered coal (60—90 mesh), or of a mixture of the coal and calcined anthracite, were carbonised at 860° in a silica crucible, and the crushing strength of the coke, as determined on a testing machine of the steelyard pattern, was plotted against percentage of inert material (cf. Badarau and Tideswell, B., 1923, 537 A). With a rate of heating such that the coal reached about 900° in 7 min., the curves exhibited sharp maxima in the neighbourhood of 30% of inert material; they gave, however, little definite information of the coking properties of the coal. With a much slower rate of heating (100° to 860° in 3 hrs.) a different type of curve was obtained with some of the coals, the coke made from the coal alone possessing the maximum strength and addition of inert material causing a rapid fall of strength to zero. The other coals still produced the first type of curve, but on further decreasing the rate of heating (100° to 860° in 3 hrs. 48 min.) all the coals examined except one (Mountain seam) gave curves

of the second type. This type of curve permits a quantitative comparison being made of the coking values of the coals, a relative measure of which is given by the area enclosed between the curve and the axis. A comparison of the curves throws light on the amount and thermal stability of the binding material in a coal. A. B. Manning.

Production of ammonia and hydrocarbons by the action of steam on lignite coke at 500°. K. A. Hofmann and H. Groll (Z. angew. Chem., 1927, **40**, 282—287).—The reaction $C+2H_2O=CO_2+2H_2$ proceeds at 500° practically to the exclusion of $C+H_2O=CO+H_2$, but with most forms of carbon the reactivity is insufficient at this temperature. It was found that lignite briquettes, coked at 500° and then treated with a mixture of steam and air to remove the gummy surface deposit, were sufficiently reactive to give considerable conversion figures at this temperature. Once the surface layer has been removed, the presence of air is injurious. The combined nitrogen in the coke is converted into ammonia in proportion to the carbon oxidised, the yield found being actually in excess of 100% owing to some catalytic synthesis from gaseous nitrogen under the influence of the ash. The ash content of the coked briquettes amounted to 10·3%, of which 28·0% was Fe_2O_3. The effect of a 5% addition of iron, manganese, nickel, and cobalt in the form of oxides to the powdered coke used was then tested. The conversion found was of the same order, and no considerable synthesis of ammonia occurred, but a notable increase in the methane content of the gas was found. Whilst an equal mixture of nitrogen and steam, without catalyst, yielded a gas containing 19% H and 0·5% CH_4, the same original gases in presence of 5% Mn gave a gas containing 13% H and 6·4% CH_4. Nickel was less effective, and iron less again. With a mixture of nickel and cobalt and prolonged treatment with steam and nitrogen the deficit in the hydrogen content of the resulting gas was greater than the production of methane, and the presence of liquid and solid hydrocarbons was noticed. The same production of hydrocarbons occurred when the coke was treated with dry hydrogen at 500°; it is therefore established that the process is independent of any intermediate formation of carbon monoxide. C. Irwin.

Ternary coal mixtures. D. J. W. Kreulen (Chem. Weekblad, 1927, **24**, 122—125).—Mixtures of varying proportions of a bituminous coal, a forge coal, and a gas coal were coked under standard conditions, and the swelling and coking properties tabulated. The influence of the gas coal on the degree of swelling is very marked. The nature of the coke given by the individual coals affords no indication of the character of the coke from the mixtures. S. I. Levy.

Colloidal properties of peat. A. Doumanski (Bull. Soc. chim., 1927, [iv], **41**, 166—185).—Peats on agitation with water yield a "hydro-mass" with the properties of a negatively-charged colloid. A highly-dispersed, stable hydrosol may be obtained from the mass if the peat is derived from *Sphagnum* residues; the suspensoid material has *d* 1·4 (approx.). This sol (as well as the original "hydro-mass") is coagulated by positively-charged hydrosols, neutral and acid salts, and especially by free acids, the valency of the cation playing the most important part (H > Al > Mg > Na). Adsorption phenomena accompany the coagulation. Alkalis and alkali carbonates peptise the hydrosol. Freezing of both the "hydro-mass" and hydrosol is accompanied by coagulation. S. K. Tweedy.

Suitability of charcoal from apricot kernel shells for the production of activated charcoal. A. Palkin (Bull. Univ. Asie centrale [Taschkent], 1926, **12**, 89—100; Chem. Zentr., 1926, II, 2939).—After treatment with mineral acids, the charcoal absorbs up to 52% of its weight of chlorine, but is less active when treated with sodium hydroxide solution, or with organic liquids, such as alcohol, benzene, or ether. The absorptive capacity is increased to 61% by steeping the charcoal in platinic chloride solution followed by gentle heating, but if it is heated strongly, the absorptive capacity falls to 45%, apparently owing to the formation of platinum carbide. The charcoal is reduced to dust less readily than wood charcoal prepared by Zelinski's process. L. A. Coles.

Determination of nitrogen in fuels. G. Lambris (Brennstoff-Chem., 1927, **8**, 69—73).—With the object of completely oxidising the traces of methane that are liable to be found in the nitrometer in the usual Dumas method, the combustion is carried out in two stages. The narrow combustion tube and connexions are first swept free from air with carbon dioxide, and the sample (about 0·5 g.) is then burnt in a current of oxygen, the combustion products passing successively over red-hot copper oxide (to oxidise ammonia) and metallic copper (to reduce nitrogen oxides) into a gas reservoir containing a concentrated solution of potassium hydroxide. Conditions are so regulated that no free oxygen reaches the metallic copper during the primary combustion, but an excess of oxygen is passed through the tube after primary combustion is complete to sweep all combustion products into the reservoir. The mixture of combustion gases and oxygen is passed from the reservoir over a glowing platinum spiral, and, finally, through a tube containing heated metallic copper (to remove final traces of free oxygen) to the nitrometer. W. T. K. Braunholtz.

Catalytic combustion. V. Union of carbonic oxide and other gases with oxygen in contact with a fireclay surface at 500°. W. A. Bone and A. Forshaw (Proc. Roy. Soc., 1927, A, **114**, 169—180; cf. A., 1906, ii, 434; 1926, 33, 250, 1109).—In most respects a fireclay surface catalyses the combination of moist carbon monoxide and oxygen in much the same way as a porous porcelain surface catalyses that of hydrogen and oxygen mixtures (*loc. cit.*), except that the catalysing power is weaker in the former case than in the latter. The rate of combination of a moist, theoretical mixture of carbon monoxide and oxygen, in contact with a fireclay surface at 500° in a "normal" state of activity, is always directly proportional to the pressure of the dry mixture, provided that the reaction product is rapidly removed from the system. The catalysing power of the surface can be highly stimulated by previous exposure to carbon monoxide at the experimental temperature, but the stimulus so imparted gradually disappears if the exciting cause is removed. A highly active surface exposed to

oxygen at the reaction temperature gradually loses its catalysing power, and becomes either "normally" active or very slightly more so. When either of the two reacting gases is present in excess, the rate of combination is proportional to the partial pressure of the carbon monoxide. Comparative experiments upon the relative speeds of the catalytic combustion of carbon monoxide, hydrogen, and methane, respectively, show that hydrogen is the most, and methane the least, amenable to the catalytic combustion. L. L. Bircumshaw.

Drainage and effluents from gas works. E. Jones (Gas J., 1927, **177**, 516—518).—The nature of the effluents from gas works is described, together with the methods of treatment of the effluent from ammonium sulphate plants before admission to the sewers, either by removal of the phenols from the ammonia liquor by means of benzol, or by subsequent bacterial treatment of the effluent. By the Bailey process, the toxicity of the effluent was reduced by evaporation of the "effluent" and "devil" liquors in two troughs arranged in a by-pass to the main flue, chimney gases being blown through the liquors. The efficiency of purification calculated on the oxygen absorption test was 33·5%. A. C. Monkhouse.

Absorption apparatus for the determination of organic vapours in air and gas. L. Bremer (Brennstoff-Chem., 1927, **8**, 77—78).—A modified form of Kattwinkel's apparatus (cf. B., 1924, 546) is described and depicted, in which alterations are made to the upper valve and connexions and to the two water cocks, with a view of eliminating leakages which are otherwise found to develop in time.
W. T. K. Braunholtz.

Determination of unsulphonated residue in petroleum spray oils. J. J. T. Graham (J. Assoc. Off. Agric. Chem., 1927, **10**, 124—130).—Sulphonation when used as an index of unsaturated hydrocarbons in oils for insecticidal sprays is an empirical procedure, as there is no sharp end-point, and no advantage is to be gained by prolonged heating and shaking. Blackening of certain oils is due to overheating rather than to carbonisation by the acid. Measurement of the unsulphonated residue in cases of excessive blackening is facilitated by addition, prior to sulphonation, of a measured volume of some other oil of known sulphonation value (*e.g.*, kerosene). In the method proposed, the oil, after gradual admixture with 38*N*-sulphuric acid, the temperature meanwhile being kept below 60°, is heated for 10 min. at 60—65°, and shaken for periods of 20 sec. at 2 min. intervals. The unsulphonated residue is measured after centrifuging for 5 min. at 1200—1500 r.p.m. H. J. Dowden.

Heat of combustion of marine animal oils. Marcelet.—See XII.

Bagasse as fuel. Hutcheson.—See XVII.

Humic acids. Fuchs and Leopold.—See XX.

Patents.

Process and apparatus for briquetting. E. B. A. Zwoyer, Assr. to General Fuel Briquette Corp. (U.S.P. 1,614,095, 11.1.27. Appl., 6.6.25).—In a process of briquetting loose materials with a liquid binder, an excess of binder is supplied from a tank to the mixer by a pipe line provided with a by-pass. A regulated amount of binder is admitted to the mixer, the excess being withdrawn and returned to the tank.
S. Pexton.

Manufacture of artificial fuel. M. F. Maginnis (U.S.P. 1,615,463, 25.1.27. Appl., 25.9.26).—Powdered or granular bituminous coal and finely-divided coke are heated and mixed with a hot solution of starch and glue, and then with hot alum solution. S. Pexton.

Production of compact [metallurgical] coke from peat. Gewerkschaft Gevenich (G.P. 435,305, 13.11.24).—Coke suitable for metallurgical use is obtained by coking peat which has been dried in the usual way, and then subjected to electro-osmosis to remove further quantities of water. L. A. Coles.

Distillation of solid fuel. P. E. H. Forsans (F.P. 609,814, 2.5.25).—The fuel falls gradually through a preheating zone, a distillation zone, and a zone for cooling the coke, in a vertical retort. L. A. Coles.

Fuel-distilling apparatus. F. E. Hobson (U.S.P. 1,614,028, 11.1.27. Appl., 26.11.23).—In a cylindrical vertical retort externally heated is suspended a concentric, louvred gas collector, which is flexible and capable of being collapsed or extended vertically. The fuel to be distilled is fed into the top of the retort, and occupies the annular space between the gas collector and the retort walls, down which it travels. S. Pexton.

Manufacture of adsorbent carbon. O. L. Barnebey and M. B. Cheney (U.S.P. 1,614,707, 18.1.27. Appl., 14.2.25).—The temperature of activating carbonaceous material with steam is regulated so that the gaseous reaction products contain more carbon dioxide than carbon monoxide. S. Pexton.

Method and apparatus for the manufacture of carbon black or lamp-black. E. B. Bird, Assr. to J. W. Trieschmann (U.S.P. 1,616,409, 1.2.27. Appl., 19.1.26).—Smoke from the combustion of carbonaceous material is passed through ducts with alternately contracted and expanded sections. The velocity of the smoke maintained in the contracted sections is sufficient to cause rapid expansion in the other sections, with deposition of the carbon black. S. Pexton.

Manufacture of decolorising carbons from vegetable materials. O. L. Barnebey (U.S.P. 1,614,913, 18.1.27. Appl., 23.3.21).—Vegetable materials are carbonised with the recovery of by-products. The carbonised material is heated in the presence of chlorine, whereby the impurities are removed, some of them being volatile, and the others are extracted as soluble chlorides. S. Pexton.

Revivifying process for carbons. F. B. Arentz, Assr. to U.S. Industrial Alcohol Co. (U.S.P. 1,616,073, 1.2.27. Appl., 25.10.24).—Carbon used in the purification of ethylene is revivified by exposure to live steam, and subsequently to a non-oxidising gas for the removal of condensed steam and odorous vapours. S. Pexton.

Process and apparatus for distilling or cracking hydrocarbons and purifying the vapours. Gray Processes Corp., Assees. of T. T. Gray (E.P. 249,871, 24.3.26. Conv., 24.3.25. Cf. E.P. 222,481; B., 1926, 230). —The process for removing unstable constituents from

hydrocarbons in the vapour phase by passage through catalysts causing polymerisation of such substances is modified by utilising a portion of the condensed vapours (simultaneously with the passage of steam) for cleansing the catalyst, condensation in the catalytic tower being so adjusted as to render the cleansing of the catalyst simultaneous with its use for purifying the vapours.
C. O. Harvey.

Method and apparatus for distilling mineral oils, tar, and the like. R. Neumann and L. Steinschneider (E.P. 257,257, 11.8.26. Conv., 22.8.25).—In a vacuum still, fired from below, when the vacuum gauge registers only 5 mm. pressure, the actual pressure at the heating surface may be as much as 100 mm. owing to the pressure exerted by the column of oil, and the oil therefore boils at a correspondingly higher temperature. In the present invention, cracking due to this cause is prevented by pumping oil from the bottom of a vacuum still through a pipe line into trays situated inside the still above the oil level, the conditions being so adjusted that vaporisation occurs only in the trays, the excess of oil overflowing back into the still. The still may be heated externally, internally by means of steam pipes, or by the insertion of a heater in the trays. The trays may be replaced by an oil distributor situated just below the level of the oil in the still. C. O. Harvey.

Distillation of hydrocarbons. A. M., G., T., and H. Offermann (G.P. 435,575, 11.6.20).—Wet steam is passed into mixtures of hydrocarbons boiling under atmospheric pressure, and the vapours are fractionated, *e.g.*, into benzol, gas oil, and machine and cylinder oil.
L. A. Coles.

Production of low-temperature tar from bituminous shale. A. Pfaff (G.P. 435,210, 6.3.20).—Fixed carbon in shale residues is burnt away as completely as possible in a continuously operated shaft furnace, the combustion gases being used for distilling fresh shale.
L. A. Coles.

Apparatus for separating oil, benzol, and similar liquids from waste water. J. Andresen (G.P. 434,795, 12.2.25).—The apparatus comprises settling and separating compartments, and the overflow of the latter is provided with a valve which is closed automatically by the lower density of the benzol–oil layer, to prevent these escaping with the water. L. A. Coles.

Process for converting or cracking hydrocarbon oils. W. E. Trent, Assr. to Trent Process Corp. (U.S.P. 1,619,896, 8.3.27. Appl., 27.9.24).—Charging stock is partially vaporised in a heat interchanger, and the vapours, having been raised to a cracking temperature by compression, are passed back to the heat interchanger to supply heat for vaporising the charging stock. C. O. Harvey.

Process and apparatus for cracking petroleum oil. G. Egloff and H. P. Benner, Assrs. to Universal Oil Products Co. (U.S.P. 1,619,921—2, 8.3.27. Appl., [A] 13.12.20; [B] 1.9.20. Renewed [A] 9.6.26, [B] 2.9.26.—(A) The oil is cracked under pressure by passage through a heated zone, and is delivered through a transfer line in a substantially liquid form to an expansion chamber. The vapours removed from this chamber at the end remote from the transfer line are subjected to reflux condensation, the condensate passing back to the transfer line and thence, after mixing with the heated oil, to the expansion chamber. (B) In an apparatus for treating oil by the above process a cylindrical vaporising chamber and still member are arranged to rotate in unison, and are fitted with non-rotating ends carrying influx and efflux tubes, the transfer line, and outlets for the discharge of vapour and residuum. C. O. Harvey.

Apparatus for treating petroleum oil. H. J. Halle, Assr. to Universal Oil Products Co. (U.S.P. 1,619,929, 8.3.27. Appl., 1.9.20. Renewed 18.9.26).—In an apparatus for cracking petroleum, a coil located in a furnace, and through which oil passes continuously, is also heated internally by an inner coil carrying flue gases, the annular oil passage between the coils being maintained by means of spirally arranged spacing lugs, which also act as baffles for the oil. C. O. Harvey.

Process and apparatus for treating oils. L. C. Huff, Assr. to Universal Oil Products Co. (U.S.P. 1,619,977, 8.3.27. Appl., 3.7.24).—The oil undergoes preliminary heating in a number of streams during its initial flow through a heating zone, and, after uniting the streams, the rate of flow during the final heating is increased. C. O. Harvey.

Process and apparatus for refining petroleum. F. A., and J. A. Milliff (U.S.P. 1,620,163, 8.3.27. Appl., 11.1.26).—Hydrocarbon vapours, obtained by heating petroleum, are cooled to a predetermined temperature and caused to expand suddenly and rotate at an accelerating speed to separate out condensate formed during the cooling process. The collected condensate communicates with the vapour mixture, the latter imposing a partial vacuum upon the former.
C. O. Harvey.

Bleaching cracked oils and oils produced by catalytic processes. A. Mailhe (F.P. 610,498—9, 15.5.25).—The oils are heated for about 1 hr. with stannous chloride under a reflux condenser, decanted off, washed with dilute sodium carbonate solution, and rectified by distillation. L. A. Coles.

Process and apparatus for removing wax from oil. S. H. Hall, Assr. to De Laval Separator Co. (U.S.P. 1,616,041, 1.2.27. Appl., 20.2.23).—Streams of waxy oil and a diluent, *e.g.*, benzine, are fed preheated into separate compartments of a well from which the two are separately withdrawn in regulated quantities and mixed. The heated product is caused to exchange its heat with the oils flowing into the well, and is finally chilled by contact with coils containing cooled brine. The wax separates from solution and is removed by centrifuging the oil. S. Pexton.

Oil composition. A. A. Somerville (U.S.P. 1,617,826, 15.2.27. Appl., 19.6.26).—The addition of 0·5—1% of diacetoneamine, triacetoneamine, or similar compound, obtained by the condensation of a ketone with a nitrogenous base, to petroleum lubricating oil, increases the stability of the latter, and renders it more suitable for use as a lubricant at high temperatures, and as a transformer oil. T. S. Wheeler.

Oxidation of paraffin hydrocarbons. I. G. Farbenind. A.-G., Assees. of M. Luther (G.P. 434,923,

19.8.22. Addn. to G.P. 405,850; B., 1925, 179).—In the prior process, temperature control is improved by the use of aluminium or its alloys as filling materials for the reaction chamber. S. S. WOOLF.

Manufacture of highly-active charcoal. O. ERNST and O. NICODEMUS, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,621,195, 15.3.27. Appl., 9.7.23. Conv., 15.7.22).—See E.P. 200,839; B., 1924, 326.

Bitumen emulsions (U.S.P. 1,615,303 and 1,616,904).—See IX.

Insulating electric currents (E.P. 244,068).—See XI.

Lubricating oils (E.P. 250,947).—See XII.

Decolorising medium (U.S.P. 1,618,149).—See XVII.

Methane (E.P. 244,076).—See XX.

III.—TAR AND TAR PRODUCTS.

Apparatus for the determination of phenol and pyridine [in tar oils]. R. KATTWINKEL (Chem.-Ztg., 1927, **51**, 159).—The apparatus is made in two sizes, a larger one for the determination of phenols and a smaller finely-graduated size for pyridine bases. It consists of a large upper bulb closed by a stopper and connected by a graduated tube to a smaller lower bulb with run-off cock. The sodium hydroxide or sulphuric acid is filled in to the zero mark, the oil added and the apparatus reversed, bringing the contents into the upper bulb where they can be agitated. C. IRWIN.

PATENTS.

Distilling tar (E.P. 257,257).—See II.

Flotation agent (E.P. 243,383).—See X.

Insulating electric currents (E.P. 244,068).—See XI.

Drying oil from coal tar (U.S.P. 1,616,321).—See XII.

IV.—DYESTUFFS AND INTERMEDIATES.

PATENTS.

Azo dyes for wool. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 243,758, 27.11.25. Conv., 27.11.24).—Azo dyes are formed by coupling sulphoarylpyrazolonecarboxylic esters with diazo compounds, dyes derived from pyrazolones containing a benzthiazole nucleus being excluded. Very level dyeings on wool are claimed. The preparation of methyl and ethyl 1-*p*-sulphophenyl-5-pyrazolone-3-carboxylates from phenylhydrazine-*p*-sulphonic acid, and of the corresponding chlorosulphotolylpyrazolone from 3-chlorotolyl-2-hydrazine-5-sulphonic acid, is described. Other pyrazolonecarboxylic esters mentioned are derived from 2 : 5-dichlorophenylhydrazine-4-sulphonic acid, phenylhydrazine-2-sulphonic acid, 2-chlorophenylhydrazine-5-sulphonic acid, 3-hydrazino-5-sulphosalicylic acid. Any diazo, tetrazo, etc. compounds may be used. [Reference is directed, in pursuance of Section 7, Sub-section 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 3373/08 (B., 1909, 239) and 765/97.] C. HOLLINS.

Production of polyazo colouring matters. L. H. FLETT, Assr. to NATIONAL ANILINE & CHEMICAL CO., INC. (U.S.P. 1,616,850, 8.2.27. Appl., 2.4.26).—Resorcinol is coupled with three molecular proportions of diazotised aromatic amines, which may or may not be different, and of which at least one contains a carboxylic or a sulphonic acid group. The products dye animal fibres and other substances yellow to orange to brown shades. *E.g.*, diazotised sulphanilic acid (2 mols.) is coupled with resorcinol (1 mol.), and the product is coupled with diazotised xylidine (1 mol.) to yield a *dye* which gives brown shades on sumac or chrome-mordanted leather, and chocolate shades on wood. T. S. WHEELER.

Dyed lacquers (Swiss P. 115,116).—See XIII.

Dyeing inks (F.P. 608,903).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Technical use of chlorine for the decomposition of raw vegetable fibres. P. WAENTIG (Papierfabr., 1927, **25**, 144—148).—A reply to criticisms by Wenzl (B., 1927, 69) of the author's measurements of the heat evolved during the treatment of vegetable fibres with chlorine water and with gaseous chlorine. In the latter process the results vary with the moisture content of the fibre, owing to the formation of hydrochloric acid which at high concentrations affects the course of the reaction. Moisture must, however, be present for the reaction to proceed, and as far as the yield and properties of the cellulose obtained are concerned there is no fundamental difference between the two processes. Wood cellulose can be isolated by the chlorine method provided that the wood is finely divided before chlorination. W. J. POWELL.

[Decomposition of raw vegetable fibres by chlorine.] H. WENZL (Papierfabr., 1927, **25**, 148—149).—The conclusions of Waentig (cf. preceding abstract) are not confirmed by the author's large-scale experiments. W. J. POWELL.

Deterioration of abaca (Manila hemp) fibre through mould action. F. B. SERRANO (Philippine J. Sci., 1927, **32**, 75—101).—Deterioration is due to the action of cellulose-digesting organisms, *e.g.*, *Aspergillus*, *Penicillium*, *Chaetomium*, the growth of which is favoured by damp, poor cleaning, long storage, inadequate ventilation, and lack of care in handling. S. I. LEVY.

Steeping process. Constituents of cotton soluble in water or dilute mineral acids, and effect of their removal on subsequent scouring. R. G. FARGHER, L. R. HART, and M. E. PROBERT (J. Text. Inst., 1927, **18**, T 29—45).—The weight lost by different cottons on thorough treatment with cold dilute mineral acids varies from $1\frac{1}{2}$% to over 4%. American, South American, and Egyptian cottons usually lose 2—3% in weight, whilst Sea Island cottons exhibit lower and many native Indian cottons higher losses. With water, the losses are generally slightly lower than with acids. In both cases they increase slowly as the temperature is raised. After treatment with dilute mineral acids, the residual mineral matter is invariably lower than 1%. Hydrochloric and sulphuric acids show no significant difference, but with acetic acid elimination is rather less thorough. Water is less effective than acids, and there usually remains from 0·15 to 0·25%, the ash alkalinity being

correspondingly high. The reduction in nitrogen content is exceedingly variable, and tends to be greater with dilute acids than with water. In the removal of reducing substances, the effects of water and of dilute mineral acids appear to be identical, the copper numbers of the grey yarns falling usually to 0·3—0·5. There is no correlation between the fall in the copper number and either the total or the non-nitrogenous organic matter removed during the steep. Within the limits set by possible tendering of the cotton, treatment with mineral acids causes no observable hydrolysis of the fat and wax, but merely liberates a proportion of the small amount of fatty acids initially present as insoluble soaps. The results are discussed in relation to the analytical control of the steep. Except when the grey material contains china clay or other insoluble inorganic substances, large-scale treatment with water or enzyme preparations should reduce the ash content to approximately 0·25%, and the ash alkalinity accordingly, whilst still lower values should characterise a successful acid steep. The copper number after an efficient steep will generally vary between 0·3 and 0·5. The value of the nitrogen content test is confined principally to cases where hydrolysis of nitrogenous constituents is suspected. Experiments on the relative tendering of grey and scoured yarns by acids indicate that the grey yarns are attacked a little more slowly. A water steep prior to the scour yields an appreciably better final result, the elimination of fat and wax being more complete, the methylene blue absorption lower, and the " white " superior. Treatment with acids is superior to treatment with water in producing these effects. The action of a number of typical enzyme preparations on cotton is almost identical with that of water under similar conditions.

J. C. Withers.

Moisture relations of cotton. Absorption of water by cotton mercerised with and without tension. A. R. Urquhart (J. Text. Inst., 1927, **18**, T 55—72).—One effect of tension during mercerisation is to decrease the moisture regain of the cotton from the value for cotton mercerised loose and therefore, presumably, its absorptive capacity generally. This effect is less pronounced and the variability of the material is less when using more concentrated alkali. The mercerisation ratio (ratio of moisture content of mercerised to unmercerised cotton) fell from 1·37 to 1·10 on applying tension when using 15% caustic soda, but only from 1·22 to 1·16 in 35% alkali. A slight tension is sufficient to bring about the full reduction, so that small variations in the tension usually applied in mercerisation are not likely to cause much variability in the absorptive capacity of the product. There were noticeable improvements in lustre, however, by increasing the tension, after the effect on the absorptive capacity had become negligible, and 15% caustic soda gave more lustrous yarn than 25%.

J. C. Withers.

Examination of the process of sizing cotton yarns on an experimental tape frame. F. D. Farrow and E. H. Jones (J. Text. Inst., 1927, **18**, T 1—24).—A tape frame is described which accommodates only about 20 ends of yarn. With this machine it is possible to perform sizing experiments covering all sorts of conditions without much risk of obtaining results which would be different from those of works experience. Experiments on (*a*) the relation between the compactness of a yarn (as governed by twist and grade of lint) and the amount of size picked up; (*b*) the sizing of yarns of different counts and (*c*) of different cottons spun to equal counts and twist; (*d*) the effects of size concentration and (*e*) viscosity; (*f*) the effect of the speed of the machine; and (*g*) the effect of the nature of the cloth covering the sizing roller and also the roller pressure were made. For the purpose of deciding many questions relating to penetration, use is made of cross-sections of the warps. stained with iodine. The results are discussed in a general review of the process of sizing in which the influences of the yarn properties (nature and extent of the surface), the size properties (concentration and viscosity), and the mechanism of the machine (roller covering, pressure, drying systems) are analysed.

J. C. Withers.

Chlorination of wool. J. B. Speakman and A. C. Goodings (J. Text. Inst., 1926, **17**, T 607—614).—Examination of the chlorination of woollen cloth, and the shrinkage in area on subsequent milling with soap or dilute sulphuric acid, led to the view that the " unshrinkable finish " obtained was due to the formation of a layer of jelly between the unattacked cortex and the cuticle of the wool by the consecutive actions of chlorine and soap or soda, and that this layer and its surrounding scales are easily removed by friction and render the " unshrinkable " wool bad for wear. An improvement in this respect can be effected by hardening the jelly layer to the unattacked part of the cortex, the best method being by chrome mordanting. The practical difficulty in the way of attaining absolute unshrinkability is that of securing uniform chlorination of all the fibres.

J. C. Withers.

Action of ammonia on wool. H. E. Farrar and P. E. King (J. Text. Inst., 1926, **17**, T 588—590).—The removal of sulphur from wool by heating with periodically replenished solutions of ammonia in sealed tubes at 50° or 70° was investigated. Sulphur was determined in the original cloth by fusion with sodium peroxide in a bomb as used for coal, and in the ammoniacal extract after oxidation with bromine water. In an extreme case the sulphur content fell from 3·9% to 2·0%, and about 14% of the wool substance was dissolved. The use of lime water, renewed daily, did not reduce the sulphur content below 3·3%.

J. C. Withers.

Action of formaldehyde on wool. H. S. Bell (J. Soc. Dyers Col., 1927, **43**, 76—79).—The amount of formaldehyde taken up by wool varies directly with the concentration of the bath up to a maximum absorption with 3% formaldehyde solution; above this with increasing concentration the absorption is practically constant. After 30 min. treatment there is no increase in the amount of formaldehyde taken up by the wool. The amount taken up varies with the volume of the bath, a result typical of absorption phenomena. A large proportion of the absorbed formaldehyde is retained on drying, and of this about 55% is not removed on treatment of the fabric with 1% neutral hydrogen peroxide. Treatment with formaldehyde renders wool less liable to damage by alkali. The absorption of formaldehyde

from the bath was followed by titration with iodine; Blank and Finkenbeiner's method (B., 1899, 79, 614) is not sufficiently accurate with very dilute solutions.

R. BRIGHTMAN.

Chemical decomposition of wool at 100°. J. L. RAYNES (J. Text. Inst., 1927, **18**, T 46—47).—Pure neutral wool containing moisture is attacked when heated at 100° in a sealed tube, ammonia being liberated. In a current of pure air, however, the decomposition is inappreciable unless the wool contains alkali. Normally scoured wool contains sufficient alkali to effect a considerable production of ammonia, especially if the wool is moist; in one case 0·105% N as ammonia was liberated in 1 hr. from a sample washed with soap, rinsed, and tested with 63% of moisture on the weight of the wool. J. C. WITHERS.

Uniformity of heavy sizing in mill practice. S. M. NEALE (J. Text. Inst., 1927, **18**, T 25—28).—Observations and records were taken in a mill concerned with the heavy sizing of dhootie warps and occasionally troubled with complaints of wide variations in cloth weight. The cause of the cloth falling short in weight was definitely traced in the size on the warp, and this to dilution of the size paste. Fluctuations in size concentration are shown to arise from condensation of the steam used for heating the size, combined with irregularity in size supply due to faulty operation of the automatic valve. Slight changes in temperature did not appreciably affect the amount of size picked up.

J. C. WITHERS.

Decomposition of cellulose in the soil. S. WINOGRADSKY (Compt. rend., 1927, **184**, 493—497).—In continuation of earlier work (B., 1926, 975) a siliceous gel, p_H 7·0, impregnated with mineral salts, provided with nitrogen in the form of nitrate, and having a filter paper spread on the surface, has been used to isolate from soils a dozen bacterial forms, which are classified in two groups—cytophaga and vibrios. The former have a marked fibrolytic action, which converts the fibre of the paper into a transparent mucilage, the colour of which—red, orange, or yellow—is characteristic. The fibrolytic action of the vibrios, however, is less pronounced, but they spread rapidly through the paper; the course of the action is followed by the colour produced during the oxidation of the cellulose. In two cases the formation of a yellow colour soon stopped without the production of any mucilage, and the experiment could then be kept for weeks without further change. In two other cases the colour was yellowish-brown, and was accompanied by much mucilage. Another vibrio, isolated from undergrowth, transformed the paper into a swollen, brown, transparent jelly. Both groups, but especially the cytophaga, cause general autolysis of the cells. The fibres swell and lose their structure, while the microbes, also swollen, retain for a time the shape of the fibres, and then gradually disappear, leaving no spores, but only a few small grains. Unlike the anaerobic bacteria, these microbes evolve no gas and produce no volatile fatty acids; on the other hand, the siliceous gel becomes alkaline owing to the absorption of the nitrate ions. In most cases the cellulose is transformed into a mucilage which is soluble in dilute alkali. J. M. GULLAND.

Alkali-cellulose. IV. E. HEUSER (Cellulosechem., 1927, **8**, 31—32).—Polemical. A solution containing 16% of sodium hydroxide has the greatest swelling action on cotton cellulose, and converts it quantitatively into the alkali-cellulose derivative, whilst for ramie, artificial silk, and wood cellulose the concentration required is about 12%. The swelling of cellulose takes place even in presence of considerable quantities of alcohol (cf. B., 1927, 40). W. J. POWELL.

Pulping of Scottish pinewood by the sulphite process. E. HÄGGLUND (Cellulosechem., 1927, **8**, 25—31).—Scottish pinewood from young trees without heartwood, or sapwood of older trees, can be utilised for the preparation of sulphite pulp, especially if a solution of magnesium bisulphite is used. The product is, however, not uniformly pulped, and still contains resin, which is dissolved to a great extent at the beginning of the boil, and is later reprecipitated on the fibre. Thus the ether extracts of the original wood and the product after 6 and 8 hrs.' boiling are 2·76, 2·0, 3·25%, respectively, the corresponding benzene extracts being 1·72, 0·16, 0·28%. Scottish pine heartwood is not suitable for use in the sulphite process, as the product is not well pulped, and contains badly-boiled wood chips with high resin content. The resin can be removed from the wood by extraction with benzene or ether, but unless the solvent is removed completely from the extracted heartwood the latter is less amenable to treatment than before extraction. This is not due to the dehydration and hardening of the cell membrane by the solvent, since if the solvent used is completely removed by washing the extracted wood with acetone before boiling with bisulphite, a white, uniformly pulped product results. The strength of the fibre is not appreciably affected by the extraction process, although the fibre from benzene-extracted wood is slightly more brittle than that from unextracted wood. Pinewood can be dried at 70° before boiling without affecting the yield or the "bromine number" of the pulp, or the strength of the fibre, except for a slightly increased brittleness as shown by the folding test. The yield of pulp in the soda process from Scottish pine heartwood is uninfluenced by extraction of the wood with benzene, but if the solvent is removed by means of acetone before boiling, the pulping takes place more rapidly and the product is more uniform.

W. J. POWELL.

Chlorine consumption of wood pulps. A. EHRENFRIED (Papierfabr., 1927, **25**, 130—131).—A modification of the Schwalbe–Sieber method for determination of the chlorine consumption value of wood pulps is proposed. After treatment for 1 hr. at 20° of 5 g. of the pulp with bleach liquor equivalent to 6% of active chlorine on the weight of the cellulose, 50 c.c. of the residual liquor are treated with 20 c.c. of 0·1*N*-arsenious acid solution and 3—5 drops of methyl orange added. The solution is treated with 10—15 c.c. of strong hydrochloric acid and titrated with 0·1*N*-potassium bromate solution. The discharge of the pink colour, due to the liberation of bromine, indicates the oxidation of the excess of arsenious acid. J. F. BRIGGS.

Universal method for converting fibroin, chitin, casein, and similar substances into the ropy-plastic state, and into the state of colloidal solution

by means of concentrated aqueous solutions of readily soluble salts, capable of strong hydration. P. P. VON WEIMARN (J. Text. Inst., 1926, 17, T 642—644).—See B., 1927, 136.

PATENTS.

Manufacture of waterproofing compositions. NAUGATUCK CHEMICAL Co., Assees. of A. F. OWEN (E.P. 251,961, 21.4.26. Conv., 7.5.25).—A water-insoluble and water-repellant saponification product of a metal (*e.g.*, aluminium, zinc, or lead) with various resins or with stearic, oleic, or palmitic acids, with or without a filler, *e.g.*, whiting, is mixed in the dry form either with an aqueous solution of a colloid, such as glue, or with water and ground in a mill. The product is stirred into a water dispersion of rubber latex (vulcanised or unvulcanised), or of any artificial rubber, and, if necessary, an oil or rubber softener in the form of a water emulsion is added. The product is specially suitable for paper. B. P. RIDGE.

Waterproofing vulcanised fibre. E. E. NOVOTNY and C. J. ROMIEUX, Assrs. to J. S. STOKES (U.S.P. 1,616,062, 1.2.27. Appl., 4.9.23).—Cellulosic material is impregnated with a synthetic resin, *e.g.*, a phenol–furfuraldehyde resin (cf. U.S.P. 1,398,146; B., 1922, 66 A), and, after immersion in water, is treated with a solution of zinc chloride, washed, heated, and pressed. T. S. WHEELER.

Washing and cleaning preparations [for textiles]. BADISCHE ANILIN- U. SODA-FABR. (E.P. 244,104, 3.12.25. Conv., 5.12.24).—Sodium *iso*propylnaphthalene-β-sulphonate (3 pts.) in water (7 pts.) is mixed with a "synthol" fraction (3 pts.) of b.p. 145—165° (*iso*butyl and higher alcohols); or an aqueous solution of a syntan (*e.g.*, formaldehyde–cresolsulphonic acid) is mixed with *cyclo*hexanol. The claims cover mixtures of aromatic (excluding hydroaromatic) sulphonic acids, having more than 10 carbon atoms in the molecule, with higher aliphatic alcohols, including alicyclic alcohols when the sulphonic acid has tanning properties. C. HOLLINS.

Manufacture of artificial light filters. C. HOLUB (E.P. 265,455, 13.7.26).—Silk or artificial silk fabric is washed with soapy water, hardened by dipping it into 50—60% formaldehyde or a dilute aqueous solution of alum, tannic acid, gallic acid, or potassium dichromate, and dried. It is then coated with a viscous colloidal substance such as gelatin, agar-agar, etc., to which a saturated solution of copper sulphate or chloride is added to colour it, and, while still wet, is exposed to formaldehyde fumes until the film coating is rendered insoluble. The filter is preserved by hermetically enclosing it in a transparent casing. B. P. RIDGE.

Artificial textile products. H. DREYFUS (U.S.P. 1,616,787, 8.2.27. Appl., 5.2.25. Conv., 29.3.24; cf. E.P. 209,125; B., 1924, 213).—Cellulose acetate or other cellulose derivative is dissolved in a low-boiling solvent, *e.g.*, formaldehyde or acetaldehyde, containing a higher-boiling precipitant, *e.g.*, benzene or alcohol, and the solution is spun into an evaporative atmosphere. A homogenous filament is obtained. T. S. WHEELER.

Weighting natural silk. R. CLAVEL (E.P. 266,640, 11.11.26. Conv., 17.9.26).—Substances which interact to form an insoluble weighting compound (*e.g.*, stannic chloride and sodium phosphate) are applied to the silk in the same bath which also contains one or more agents which hinder precipitation, *e.g.*, acids, acid salts in relatively high concentration, or such protective colloids as glue, gelatin, soap, etc. The weighting process is thereby considerably shortened and intermediate washings of the fabric are avoided. The colloid protects the silk from injurious acid attack, and may be applied separately before weighting. If necessary, two or more weighting baths may be used, without intermediate washing of the fabric, the latter being finally squeezed and treated with an alkaline solution such as ammonium carbonate to which substances containing tannin may be added for subsequent dyeing purposes. B. P. RIDGE.

Production of weighted artificial silk. E. CADGENE, Assee. of G. RIVAT (E.P. 259,899, 11.1.26. Conv., 15.10.25).—Cellulose acetate silk is treated with a gelling agent (15—20% formic or acetic acid), drained, steeped in a heavy-metal salt solution, *e.g.*, stannic chloride, washed, again gelled, and the tin fixed by means of a sodium phosphate bath at 40—45°; finally, the material is washed and soaped. Alternatively, gelling and weighting may take place simultaneously by the addition of acetic acid to the stannic chloride solution. From 30 to 100% weighting may thus be obtained, the material remains soft and lustrous, and has an increased affinity for certain dyestuffs. B. P. RIDGE.

Degreasing raw wool etc. A. M. BRUCKHOFF (E.P. 266,436, 23.11.25. Addn. to E.P. 241,314; B., 1926, 9). —Instead of acetone, other mild grease solvents, such as methyl acetate, methyl alcohol, etc., or mixtures thereof (*e.g.*, wood spirit) which are soluble in water in the ratio of at least 1 : 10 by wt. are used. Raw wool or wool waste containing fat or mineral oil is dried until it contains only 2—3% of moisture, extracted with the solvent at a temperature below its b.p., and subsequently washed with water, the solvent and dirt being thereby removed. Potassium compounds are recovered from the washing water. B. P. RIDGE.

Manufacture of pulp or fibrous material. R. A. MARR, and RAMAR SYNDICATE, INC. (E.P. 266,168, 24.4.26).—Cellulosic or ligneous material is digested with 0·05—3·5% solutions of the sulphates, chlorides, or nitrates of the alkali, alkaline-earth, or earth metals (except zinc), or combinations of these, at 30—120 lb. excess pressure per sq. in., the intercellular cementitious material being thus rendered friable and removable by washing after the product has been crushed. The product is finally pulped by passing through a beater. The cementitious material is filtered off and pressed into moulds, or may be mixed with resinous or sizing materials before being moulded. B. P. RIDGE.

Decomposition of cellulosic materials. G. MEUNIER (F.P. 609,191, 28.11.25).—Wood or straw is decomposed when warm by means of steam (mixed, if necessary, with other vapours or gases), acid, and neutral solutions, and, if required, oxidising or reducing agents. The gaseous products, besides lignin, acetic and formic acids, furfuryl and methyl alcohols, acetone, and ferment-

able sugar, are recovered. The treatment gives results equal to those obtained by means of mineral acids. B. P. Ridge.

Treatment [precipitation] of cellulose. K. B. Heberlein (F.P. 610,417, 29.1.26).—Cuprammonium cellulose solutions are subjected to electrolytic treatment to remove the metallic constituents. L. A. Coles.

Production of cellulose product. H. H. Clark, Assr. to Clark Fibre Products Corp. (U.S.P. 1,618,572, 22.2.27. Appl., 8.3.24).—Cellulose fibre is saturated with a mixture of 100 pts. by wt. of an aqueous solution of zinc chloride, *d* 2 (approx.), and 3—10 pts. of phosphorus oxychloride. The mass is compacted and the liquid gradually removed from it by successive washings in progressively weakened baths of the same solution. The product (*d* over 1·25), containing about 0·1% of added compounds, is semi-plastic under stress, and has useful mechanical properties. B. P. Ridge.

Manufacture of cellulose acetate. L. A. Levy and O. Silberrad (E.P. 265,267, 7.8.26).—Cellulose is treated with a mixture of acetic acid and acetic anhydride in the presence of chromium as a catalyst. The metal may be present as acetate, sulphate, butyrate, or in any other form capable of producing salts with the acetylating mixture. B. P. Ridge.

Coating articles with cellulose acetate. F. T. Ridley (E.P. 265,257, 28.9.25).—Cellulose acetate in the form of sheets or tubes is treated with an acetone-water or an alcohol-water solution until it is of the consistency of indiarubber, stretched over the object to be covered, and allowed to harden. The former solution contains, by volume, 75—55 pts. of water, 25—45 pts. of acetone, and sufficient ether to saturate it, the latter containing about 70 pts. of water, 30 pts. of alcohol, and 15 pts. of ether, a small quantity of soap being added in each case if desired. Solutions which have deteriorated through use are revived by heating them to below the b.p. of the lowest-boiling constituent. B. P. Ridge.

Cellulose acetate products. I. G. Farbenind. A.-G. (Swiss P. 115,676, 8.4.25. Conv., 29.4.24).—Material capable of acting as a solvent or swelling agent for cellulose acetate, *e.g.*, acetone or formaldehyde, is added to the precipitation bath. L. A. Coles.

Manufacture of benzylcelluloses. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 265,491, 1.10.26).—Crude benzylcellulose is purified by mixing with a water-soluble salt (preferably of the alkali or alkaline-earth metals, *e.g.*, sodium chloride), extracting the impurities with a solvent, or a succession of different solvents, *e.g.*, benzene, ethyl alcohol, acetone, and finally washing out the residual salt with water. Alternatively, the impurities are only partly removed before washing out the salt and the remainder extracted afterwards. B. P. Ridge.

Process and apparatus for making paper etc. C. H. Allen (U.S.P. 1,620,860, 15.3.27. Appl., 11.1.26).—The consistency of certain stock ingredients, such as ground wood pulp, is automatically controlled and maintained near the source of supply. The material is continuously delivered to a common stream where mixing occurs, and a continuous flow is maintained direct to the paper-making machine. B. P. Ridge.

Manufacture of decorated paper, or similar material. V. Lefebure (E.P. 265,334, 7.12.25).—A mixture of a phenol- or carbamide-formaldehyde synthetic resin (in the form of a solution, syrup, or powder) with a colouring matter is printed or stencilled on to the entire surface of the paper, which may or may not have been previously impregnated with a solution of the resin; the paper is dried below 100°, pressed, and finally heated at a higher temperature to cure the resin. Such paper may be cured on to a backing of asbestos cement. B. P. Ridge.

Paper sizing. J. A. De Cew, Assr. to Process Engineers, Inc. (U.S.P. 1,621,399, 15.3.27. Appl., 11.4.23).—Rubber emulsions are coagulated on to paper-making fibres while preparing the stock for the paper machine by mixing the emulsions with the fibres in the presence of precipitated aluminium hydroxide. B. P. Ridge.

Non-inflammable lacquers for fabrics (E.P. 243,031).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Patent.

Production of Aniline Black reserves by means of vat dyes. E. Zeidler (Austr. P. 103,622, 29.12.23).—The vat dye is printed according to the soda-potash-hydrosulphite process with addition of zinc oxide and sodium acetate on the unprepared goods, which are then treated in an air-free Mather-Platt machine with wet steam until the vat dye is completely vatted, and are finally entered into the Prudhomme Aniline Black bath, to which may be added *p*-phenylenediamine. The reserves are fast to light and washing. C. Hollins.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

By-product hydrochloric acid. Its use in the extraction of phosphate rock. E. J. Fox and C. W. Whittaker (Ind. Eng. Chem., 1927, **19**, 349—353).—American-produced potash is largely potassium chloride. As the sulphate is preferred for some purposes and commands a higher price per unit of K_2O, it is proposed to convert the chloride into sulphate and utilise the hydrochloric acid produced in the extraction of phosphate rock. Florida pebble phosphate has the composition $Ca_3P_2O_8,3CaO$. On dissolution in hydrochloric acid a mixture of phosphoric acid and monocalcium phosphate is produced, the maximum concentration of phosphorus in solution occurring when half of it is in each form. To convert the solution into dicalcium phosphate lime must then be added. It is calculated that 100 tons of rock require the addition of 57 tons of hydrogen chloride and 33 tons of calcium carbonate, the products being 60·5 tons of dicalcium phosphate and 80 tons of calcium chloride. Phosphate rock cannot be used to neutralise the acid solution, the rate of reaction being very slow. If calcium carbonate is used, part of the phosphate is converted into the tricalcium salt. It is therefore proposed to precipitate the solution with milk of lime.

Experiments in support of these conclusions are described. The milk of lime precipitate contains about 40% P_2O_5, only traces of which are citrate-insoluble. C. IRWIN.

Instability and explosiveness of bleaching powder and calcium hypochlorite. H. KAST and L. METZ (Z. Elektrochem., 1927, **33**, 21—39).—The following materials were examined: (1—3) commercial bleaching powders containing respectively 36·5%, 32·5%, and 20% of active chlorine; (4) chemically pure bleaching powder, free from carbonate, containing about 42% of active chlorine; and (5) "Perchloron," a technical calcium hypochlorite containing 68·1% of available chlorine. The free lime (including carbonate) in these five materials was about 11·7, 20·6, 34·7, 14·0, 13·6% CaO respectively. The behaviour of the materials was examined under the following conditions: (A) Heated in glass tube over flame. (B) Heated in glass tube for 1 hr. at various constant temperatures between 56° and 135° and the amounts of chlorine and oxygen evolved determined: (1) and (4) are most easily decomposed, yielding much more chlorine than oxygen; (2) decomposes less but yields a larger proportion of oxygen: (5) is still more stable and gives more oxygen than chlorine; (3) is the most stable and evolves oxygen only. (C) Heated at constant temperature of 79° for long periods: only (1) gives notable amounts of chlorine, but all give off oxygen, the amount increasing in the order (3), (1), (5), (2). (D) Effect of moisture on decomposition during 1 hr. at various constant temperatures: the rate of decomposition is increased, particularly with (5), which now evolves a large proportion of chlorine. (E) Carbon dioxide passed through a column of the material at room temperature: no oxygen was obtained in any case; the amount of chlorine obtained from (1) was generally large, but varied enormously with different samples; (4) gave less chlorine than was obtained from any sample of (1); (2) gave intermediate amounts, but (3) gave none. With moist carbon dioxide, decomposition was much more considerable for (1) and (2), and was detectable for (3). Hence exposure to the atmosphere can greatly accelerate the decomposition of bleaching powder. (F) Sensitiveness of mixtures of bleaching powder or Perchloron with benzene, tetralin, mineral oil, sawdust, wood charcoal, iron oxide, or iron powder, towards rise in temperature, exposure to an open flame, and subjection to mechanical shock, was examined. Mixing a large proportion of tetralin with (1) causes vigorous action; a marked rise in temperature also occurs on mixing (1) with wood charcoal or (5) with tetralin in suitable proportions. Mixtures of bleaching powder with the various materials specified are generally not sensitive to an open flame (a combustible addition agent merely burns off), nor do they exhibit any definite explosion phenomena when subjected to a hammer blow. Perchloron mixtures, however, are very sensitive to flame; mixtures with tetralin or mineral oil burn like black powder, and also behave as weak explosives when struck by a drop hammer. Detonation in a lead cylinder or an iron tube shows that neither bleaching powder, Perchloron, nor Perchloron + 8% of tetralin is really explosive, but Perchloron + 8% of mineral oil has a notably explosive character, though not as great as that of black powder. Mixtures of (1) with wood charcoal or with tetralin in large proportions undergo vigorous decomposition and the gases produced soon burst a closed container. It is concluded that neither high-grade bleaching powder nor Perchloron is to be regarded as an explosive, but merely as an oxygen carrier comparable with potassium nitrate and less active than potassium chlorate; they differ from these salts in giving up their oxygen at lower temperatures, though at ordinary temperatures they are fairly stable, and their stability is increased by the presence of free lime. In spite of its higher available chlorine content, Perchloron is safer to transport than bleaching powder, which decomposes slowly at ordinary temperature, but where contact with organic matter is possible, Perchloron is the more dangerous. H. J. T. ELLINGHAM.

Wear on copper preheater tubes in potash plants, and the detection of traces of copper in concentrated alkali chloride solutions. G. JANDER and H. BANTHIEN (Z. angew. Chem., 1927, **40**, 287—288).—Brine for the dissolution of potassium chloride is usually preheated in copper tubes surrounded by steam. The wear on these tubes is considerable, and the effect of electrical protection uncertain. In a particular works it was calculated that the solution leaving the tubes must contain 0·3—0·5 mg. Cu per litre. This copper remained in solution after the liquor was crystallised, was in the cupric state, and was scarcely precipitable by hydrogen sulphide. It was determined by precipitating, along with iron, with potassium ferrocyanide, decomposing with sulphuric acid, and proceeding by Classen's method. During the passage of the liquor through the copper tubes ferric iron is reduced to the ferrous state and the reaction $2\,Fe^{+++} + Cu = Cu^{++} + 2\,Fe^{++}$ probably occurs. The corrosion might be avoided if the solution was, previous to its contact with the copper, reduced by passing over iron borings or scrap copper. C. IRWIN.

Industrial preparation of fluorine. P. LEBEAU (Bull. Soc. d'Encour., 1927, **126**, 15—35).—Types of cell used for the preparation of fluorine since its discovery are reviewed. The earlier ones employed a platinum anode, which was rapidly corroded, in the electrolysis of hydrogen fluoride at low temperatures. Later ones used Acheson graphite as anode and worked with fused salts, such as potassium hydrogen fluoride. In this case carbon tetrafluoride is formed, the amount increasing with the temperature, the current density, and the *E.M.F.* used. The salt KF,3HF melts at 56° and KF,2HF at 70°, whilst the m.p. of KF,HF is very much higher. It is, therefore, advantageous to use the first-named salt in the fused state, as some acid may be lost without necessitating any considerable rise of temperature. Under these conditions a nickel anode is not attacked, and is employed in the author's apparatus. The cell is of copper, the walls forming the cathode. The nickel anode in the form of a cylindrical bar, the lower part of which is hollow, is supported within a copper bell which dips below the surface of the bath, and the lower part of which is perforated. When electrolysis commences, this copper bell becomes immediately covered with a coating of copper fluoride, and fluorine is henceforth produced only at the nickel. The

bell thus serves as the diaphragm. The evolved gas is removed from an outlet in the bell, and with 2·1 amp. and 30 volts the yield is 845 c.c./hr. The acidity of the bath is maintained by addition of hydrogen fluoride to the outer portion from a copper cylinder of the liquefied acid. The initial electrolyte, KF,3HF, is prepared by weighed addition of hydrogen fluoride to KF,HF in a copper bottle. The apparatus works smoothly, and yields of above 96% were obtained at 56°. With increase of temperature the efficiency gradually falls. The *E.M.F.* used need not exceed 8—10 volts. As anhydrous hydrogen fluoride can be easily prepared by heating dry potassium hydrogen fluoride in an apparatus entirely of copper, the laboratory preparation of fluorine is now relatively simple. The extension of the method to industry only depends on applications for the element, which may include its employment in the preparation of ozone, or, in the form of metallic perfluoride, as an oxidising agent in organic chemistry. C. Irwin.

Twenty-five years of progress in the cyanamide industry. W. S. Landis (Trans. Amer. Electrochem. Soc., 1927, **51**, 79—86).

Ammonia from lignite coke. Hofmann and Groll.—See II.

Behaviour of oxides in salt solutions. Feitknecht.—See IX.

Patents.

Manufacture of sulphuric acid. R. G. y de Sotto (E.P. 265,857, 20.7.26).—An increased yield of some 30% of acid is obtained if the gases containing sulphur dioxide, prior to treatment in the Glover tower or catalyst chamber, are subjected to the action of radium emanations, and also to the influence of ultra-violet rays produced by a quartz lamp. An electrical resistance maintains a temperature of 900—1200° round the tube containing the radium or radium salt. W. G. Carey.

Apparatus for the manufacture and delivery of hydrocyanic acid gas. H. Lainé (E.P. 262,423, 30.11.26. Conv., 5.12.25).—Hydrocyanic acid gas is produced safely in an apparatus consisting of a circular vessel containing acid or a reactive powder surmounted by a cylindrical vessel fitted with a gas outlet and with baffles inside to retain particles of acid which tend to be drawn along by the gases evolved. A reservoir containing a cyanide solution is situated above the cylindrical vessel, the solution being admitted to the acid container by an external tube fitted with a needle valve.
W. G. Carey.

Production of phosphoric acid from moist producer gas containing phosphorus compounds. W. Kyber (G.P. 434,922, 29.1.26).—Sufficient air or oxygen is added at 100—1000° to the gas to convert any phosphine present into phosphorous acid, which is then heated at 400° to convert it into phosphoric acid and phosphine, the latter being returned to the process.
L. A. Coles.

Production of phosphoric acid and acid phosphates. Chem. Fabr. Bernburg Dr. H. Wagner & Co., and A. Finkelstein (G.P. 434,985, 19.6.25).—Dialkali phosphates or alkali pyrophosphates are converted by electrolysis into alkalis and trialkali phosphates on the one hand, and into phosphoric acid and monoalkali phosphates on the other. L. A. Coles.

Field tube evaporator for alkali hydroxide solutions. G. Sauerbrey Maschinenfabr. A.-G. (G.P. 435,266, 10.8.24).—The individual tubes in each group are connected in series in such a manner that the steam passes through the head to the far end of the tube and then back to the head, whence it passes to the next tube of the series. The tube heads in each series are connected with those of the series above by tubes for conveying away condensed water. A maximum heating effect is attained by widening the ends of the steam inlets. L. A. Coles.

Catalyst for ammonia synthesis. L. H. Greathouse (U.S.P. 1,618,004, 15.2.27. Appl., 31.3.22. Cf. F.P. 436,472; B, 1912, 432).—Ferric oxide or manganese dioxide is mixed with aluminium powder, just sufficient when the thermit reaction is initiated to fuse all the oxide, no substantial portion of which is reduced. When cold the hydroxide is ground and reduced in a current of hydrogen. The metal so obtained is an active catalyst for the synthesis of ammonia. T. S. Wheeler.

Purifying salt [potassium alum] crystals from iron contaminants. H. P. Bassett, Assr. to Electro Co. (U.S.P. 1,616,250, 1.2.27. Appl., 27.3.25).—Potassium alum crystals which are contaminated with adhering ferrous and ferric sulphates are purified by washing first with sodium chloride solution, then with water. T. S. Wheeler.

Production of potassium nitrate from crude potassium salts. Wolff & Co., and F. Frowein (G.P. 435,155, 31.3.25).—Blanc fixe and magnesium nitrate solution for use in the production of potassium nitrate are obtained by the action of 47—49% nitric acid upon a suspension of barium carbonate in magnesium sulphate solution. The carbon dioxide liberated and sodium salts remaining after extraction of the potassium salts in the crude salts are used for the manufacture of sodium carbonate. L. A. Coles.

Production of potassium chloride from carnallite. B. Stollberg (G.P. 435,212, 25.10.21).—Carnallite is extracted with hot liquor in two stages such that in the first only magnesium chloride and in the second practically only potassium chloride is dissolved. The artificial carnallite which crystallises out on cooling the liquor obtained in the first stage is treated with the second extract to dissolve out the magnesium chloride, and the solution thus obtained is used in the first stage of the extraction of fresh material. L. A. Coles.

Production of anhydrous sodium sulphate. J. W. Hill (Can. P. 261,891, 22.6.25).—Anhydrous sodium sulphate is precipitated by heating sodium sulphate solution saturated at about 32·4°. L. A. Coles.

Production of sodium sulphite and ammonium chloride. Chem. Fabr. Kalk G.m.b.H., and H. Oehme (G.P. 434,983, 7.7.25).—Sodium sulphite formed by the double decomposition of ammonium sulphite and sodium chloride is crystallised as completely as possible from the hot solution and removed, after which the residual sodium sulphite in the solution is wholly or partially converted into sodium bisulphite, and the solution cooled to below 35°, sodium chloride being added to it.
L. A. Coles.

Saturator for the production of ammonium

sulphate. L. Chevalet (F.P. 610,936, 15.12.25).—The apparatus consists of an inclined hemicylinder, the inner walls being of sheet lead of which the upper and lower portions are joined by bolts. The ammonium sulphate passes through the apparatus in counter-direction to the ammonia. L. A. Coles.

Production of alkali sulphates and ammonium sulphate, or their double salts. F. Stein (F.P. 610,906, 20.10.25. Conv., 20.10, 11.11, and 23.12.24).—Magnesium sulphate and calcium sulphate and, if desired, an alkali salt, such as the sulphate or chloride, are treated with ammonia and carbon dioxide.

L. A. Coles.

Manufacture of basic calcium hypochlorites. S. Urano and S. Imai (E.P. 266,180, 18.5.26).—Slaked lime is added to a clear aqueous solution of bleaching powder, and the solution, containing about 3% of available chlorine, is evaporated *in vacuo* to a concentration of 6·5% of available chlorine, when dibasic hypochlorite containing 45% of available chlorine separates out. The monobasic salt is obtained if the original solution is concentrated to 10% of available chlorine.

W. G. Carey.

Manufacture of pure calcium hypochlorite. S. Urano and S. Imai (E.P. 266,181, 18.5.26).—Dibasic calcium hypochlorite is agitated with water for 1 hr., and the insoluble calcium hydroxide separated by filtration, the filtrate being evaporated *in vacuo* until calcium hypochlorite crystals are obtained, which are dried *in vacuo*. W. G. Carey.

Production of calcium arsenate. H. K. Moore, Assr. to Brown Co. (U.S.P. 1,619,267, 1.3.27. Appl., 14.2.25).—Finely-divided arsenious acid is added to a solution containing calcium hydroxide and a substance capable of evolving nascent oxygen, the whole being constantly agitated. H. Royal-Dawson.

Manufacture of copper sulphate from precipitates containing copper. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 266,237, 23.9.26).—Copper sulphate free from chlorine for electrolysis is obtained by dissolving the basic copper chloride precipitate in sulphuric acid, concentrating until the solution contains not more than 25% Cl, and crystallising the copper sulphate. The crystallisation of pure copper sulphate may also be effected in solutions containing not more than 10—15% Zn, or zinc sulphate may be added equivalent to the basic copper chloride, with the formation of zinc chloride and copper sulphate. W. G. Carey.

Method and apparatus for recovering and regenerating leaching solutions. H. E. Wetherbee, Assr. to R. F. Grant and H. M. Hanna (U.S.P. 1,615,970, 1.2.27. Appl., 10.5.24).—The ferrous sulphate solution, obtained by leaching copper ores with ferric sulphate and precipitating the extracted copper with scrap iron, is treated with sodium hydroxide solution, the precipitate oxidised with air, and treated with sulphuric acid to yield ferric sulphate solution. The sodium sulphate solution obtained is electrolysed to give sodium hydroxide and sulphuric acid. T. S. Wheeler.

Manufacture of aluminium chloride. F. W. Hall, Assr. to Texas Co. (U.S.P. 1,619,022, 1.3.27. Appl., 30.12.21).—An alumina–carbon mixture containing excess of carbon is passed through a retort the mixture is treated with chlorine, and the aluminium chloride vapours are continuously removed from the retort. The excess carbon is continuously discharged into a chamber communicating with the retort where combustion takes place, the resultant hot gases heating the retort. W. G. Carey.

Manufacture of chromates and manganates. Comp. Gén. des Produits Chimiques de Louvres, and P. Pipereaut (E.P. 255,078, 8.7.26. Conv., 8.7.25).—Alloys of iron with chromium and with manganese containing about 6% C, obtained by treating the ores in electric furnaces in presence of charcoal, are pulverised to pass 100-mesh and mixed in a pan or furnace with alkali or alkaline-earth carbonates and an oxidising agent, *e.g.*, sodium nitrate. The reaction is started by heating the furnace or by internal ignition, when self-combustion takes place, the reaction being completed and the molten mass stirred by blowing in air or oxygen. The mass is tapped off and dissolved in water.

W. G. Carey.

Producing metal compounds. A. F. Meyerhofer (E.P. 265,880, 28.5.26. Addn. to E.P. 245,719; B., 1926, 1013).—The method of producing metal compounds by the decomposition of a silicofluoride or borofluoride, described in the prior patent, is modified so that the sparingly soluble fluorides of silicon or boron are not used to produce the original silicofluoride or borofluoride, but are used to produce some other silicofluoride or borofluoride, such as the zinc compound, from which the original complex fluoride is obtained by treatment with a salt containing the required base. W. G. Carey.

Production of phosphorus, phosphorus pentoxide, and phosphoric acid. I. G. Farbenind. A.-G. (F.P. 610,520, 2.2.26. Conv., 6.2.25).—The gases issuing from retorts for the manufacture of phosphorus pass through a filter chamber maintained at a temperature above the dew point of phosphorus, in which they are subjected to electrical treatment if necessary.

L. A. Coles.

Production of ozone or ozonised oxygen. H. Filz (G.P. 435,265, 14.1.23).—The gas is formed by the slow combustion of mixtures containing salts with a high oxygen content, such as chlorates or nitrates, metal oxides, material capable of supporting and retarding combustion, binding agents, and coniferous resins with a high terpene content, moulded into a porous mass or packed in suitable containers. L. A. Coles.

Process and apparatus for the manufacture of concentrated carbon dioxide. V. Drewsen, Assr. to West Virginia Pulp & Paper Co. (U.S.P. 1,619,336, 1.3.27. Appl., 12.10.21).—Carbon dioxide in kiln or other gases is absorbed in sodium carbonate liquor in a connected series of pressure absorption tanks having agitators, the sodium bicarbonate thus formed is centrifugally separated, and the carbon dioxide recovered from a heated evaporating pan provided with a reflux condenser. W. G. Carey.

Manufacture of porous adsorbents. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 266,133, 22.2.26).—Natural or artificial zeolites or slags are treated with acids to produce a jelly, or a sol which subsequently

forms a jelly, which, after complete or partial drying, is washed and gradually heated to about 220°.
W. G. CAREY.

Manufacturing nitric acid and its salts. G. KASSNER (U.S.P. 1,616,900, 8.2.27. Appl., 24.3.24. Conv., 29.3.23).—See E.P. 213,571; B., 1925, 9.

Lining for alkali recovery furnaces (U.S.P. 1,619,785).—See IX.

VIII.—GLASS; CERAMICS.

Tentative specifications for clay firebrick for malleable furnaces with removable bungs and for annealing ovens. AMERICAN REFRACTORIES INSTITUTE (Tech. Bull., 1926, No. 3). CHEMICAL ABSTRACTS.

Systems of oil burning and their possible application to the potteries. F. L. BOLT (Trans. Ceram. Soc., 1926, **25**, 263—288).

PATENTS.

Refractory articles. ALUMINUM CO. OF AMERICA, Assees. of B. T. HORSFIELD (E.P. 262,403, 2.11.26. Conv., 7.12.25).—Hollow globules of a fused refractory oxide low in silica, *e.g.*, alumina or a mixture of alumina with a small proportion of other refractory oxides, are moulded under pressure with the addition of a small proportion of a suitable binder, preferably containing a relatively large proportion of the oxide forming the globules, *e.g.*, sodium or calcium aluminates. The product, after drying and firing, has a high thermal resistance and m.p. with good mechanical strength at high temperatures.
B. W. CLARKE.

Manufacture of refractory articles. CARBORUNDUM CO. LTD. From CARBORUNDUM CO. (E.P. 266,165, 9.4.26).—A porous grog is made by burning a mixture of pure kaolin clay and an organic filler, *e.g.*, wood flour, and, after grinding, 4 pts. of this porous aggregate, 1 pt. of kaolin clay, and 1 pt. of wood flour are moulded, dried, and burnt to form porous ceramic articles capable of resisting shrinkage at high temperatures.
B. W. CLARKE.

Sheet abrasive material. U.S. SAND PAPER CO., Assees. of H. JOSEPH (E.P. 252,165, 11.5.26. Conv., 12.5.25).—A flexible sheet abrasive material comprises a waterproofed flexible backing, with a suitable abrasive secured by a waterproof, varnish-like adhesive, both backing and adhesive being fireproofed with suitable agents, *e.g.*, triphenyl phosphate or ammonium sulphate.
B. W. CLARKE.

Arc shield. C. T. EVANS, Assr. to CUTLER-HAMMER MANUF. CO. (U.S.P. 1,617,515, 15.2.27. Appl., 20.4.21).—A shield of the requisite electric and heat resistance, which is not liable to crack or spall, is made by bonding silicon carbide (92%) with a semi-refractory clay (8%) into the desired form and burning. F. R. ENNOS.

Magnesite refractory. U.S. METALS REFINING CO., Assees. of A. MARKS (U.S.P. 1,616,055, 1.2.27. Appl., 8.6.26).—See E.P. 244,391; B., 1926, 586.

Electric furnace for melting glass (E.P. 249,554).—See XI.

IX.—BUILDING MATERIALS.

Behaviour of difficultly soluble metal oxides in solutions of their salts. II. Magnesium oxide cements. W. FEITKNECHT (Helv. Chim. Acta, 1927, **10**, 140—167; cf. B., 1927, 73).—The desiccation curves of the solid masses obtained by mixing magnesium chloride solutions with magnesium oxide have a point of inflexion corresponding with the presence in the partially dried mass of 6 mols. of water per mol. of magnesium chloride in addition to the water required to convert the oxide into hydroxide. For the crystallised oxychlorides the corresponding figure was 4·4—6·7. On rehydration, those specimens take up most water which contained most originally. If the solid cement is immersed in a $5N$- or $8N$-magnesium chloride solution, magnesium chloride is taken up relatively rapidly at first, then more slowly, the rate of absorption in this latter stage being the more rapid the less the amount of chloride originally present in the cement. After some time fine crystals appear near the cement, indicating that the chloride-oxide mixture contains even after setting unchanged oxide, which first dissolves in the concentrated chloride solution and then separates out as oxychloride. The smaller the amount of water used to mix the cement, the greater is its hardness when set, as measured by the load necessary to break a cylinder of it supported horizontally at its two ends. The concentration of the magnesium chloride used to mix the cement must exceed a certain minimum value, which depends on the physical state of the oxide used, for setting to occur at all. With increase in the concentration beyond this value, the hardness of the cement increases at first rapidly and subsequently more slowly. For a given composition of the initial mixture, the hardness of the cement varies considerably with the fineness and mode of preparation of the oxide; in general the fine-grained oxide prepared from the oxalate yields the hardest masses. With increasing temperature, the minimum chloride concentration which will produce hardening increases. The hardening powers of the magnesium salts decrease in the order: chloride, bromide, sulphate, nitrate. On desiccation at the ordinary temperature the hardness increases slowly at first, then, when most of the water has been lost, much more rapidly. Rehydration causes the hardness to fall below its initial value. On dehydration at higher temperatures, the hardness commences to decrease again after a certain point, which, however, corresponds with no particular stoicheiometric composition of the mass. Microscopical examination indicates that setting is not due to the formation of crystalline oxychlorides, but to the chloride solution causing the grains of oxide to swell, owing to some chemical change in their interior, until they come into such close contact that they cohere. The effect of various salt solutions on the hardened cement has been examined. Ferric, aluminium, stannic, and cupric chlorides dissolve it to form colloidal solutions. Zinc and nickel chlorides harden it, whereas calcium and barium chlorides soften it. R. CUTHILL.

PATENTS.

Material for lining alkali recovery furnaces. J. BEVERIDGE (U.S.P. 1,619,785, 1.3.27. Appl., 7.11.25).—Concrete is made from soapstone in broken pieces

and Portland cement, excess of the former being present. W. G. Carey.

Manufacture of sheet material. A. E. Hills (E.P. 266,125, 17.2.26).—Sheet material is prepared by incorporating an organic colloid, *e.g.*, rubber, with a mixture of Portland cement and asbestos fibre, together with a solvent and fillers, *e.g.*, ground chalk, if desired. B. W. Clarke.

Waterproof coatings and damp courses for buildings and structures. C. N. Forrest (E.P. 266,224, 25.8.26).—A loosely-woven, fibrous, textile fabric, impregnated with a molten mixture of blown asphalt and a suitable petroleum oil, is described. B. W. Clarke.

Production of bituminous emulsions. L. Kirschbraun (U.S.P. 1,615,303, 25.1.27. Appl., 21.11.29).—Bitumen in a fluid state is added to a suspension of clay in water. The mixture is agitated and maintained at such a temperature that the bitumen will be dispersed without coalescence. S. Pexton.

Production of [bitumen] emulsion. L. Kirschbraun (U.S.P. 1,616,904, 8.2.27. Appl., 7.12.25).—Bitumen emulsions of the type described in U.S.P. 1,302,810 and 1,517,075 (B., 1919, 494 A; 1925, 65) can be rendered more fluid by vigorous agitation. T. S. Wheeler.

Electric furnace for cement production (E.P. 249,554).—See XI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Second experimental report to the Atmospheric Corrosion Research Committee (British Non-Ferrous Metals Research Association). W. H. J. Vernon (Faraday Soc., March, 1927, advance proof, 113—183).—In atmospheric "indoor" tests on the corrosion of copper, the influence of purity of the metal is negligible compared with the nature of the atmosphere and the metal surface. Tarnishing of copper is initiated by atmospheric sulphur, equivalent in concentration to more than 1 vol. of hydrogen sulphide in 600 million vols. of air, and, when once started, the thickness of tarnish follows a parabolic relationship with time irrespective of later considerable fluctuations in the sulphur content of the atmosphere. If, at the commencement, sulphur is present in less than the tarnishing concentration, a thin oxide film is formed which protects the metal from subsequent increase in the sulphur content of the atmosphere. Close contact of copper with smoke from cellulose produces considerable immunity from tarnishing. Oxide corrosion of zinc follows a linear form and suggests a granular structure of the corrosion film which has been corroborated under the microscope. In an atmosphere of low relative humidity, both 70/30 and 60/40 brass simulate the behaviour of copper for a considerable time, but later revert to the straight-line curve characteristic of zinc, the latter occurring wholly in a more humid atmosphere. The first attack is chiefly directed upon the α-constituent, which tarnishes, and subsequently pitting of the β-constituent occurs. Low-temperature oxide films have relatively little effect upon the tarnishing of brass, though films produced at higher temperatures exerted some protective influence, as did also treatment with lanoline. Progress of corrosion of lead and aluminium is at first parabolic, quickly retarding until after 10—14 days the oxide film has ceased to thicken. In an ordinary room atmosphere of low humidity, as obtains under conditions of artificial heating, rusting of iron is controlled entirely by the presence or absence of solid impurities in the atmosphere, and rusting may be inhibited by either filtering the air or screening the specimen behind a single thickness of muslin. Under such conditions iron develops a protective film analogous to other metals, which afterwards definitely resists attack for a time and then breaks down locally with accelerating effect. In a saturated atmosphere, the presence of suspended solid particles is not necessary to start rusting which accelerates from the start. Open-air exposure tests showed the following order for erosion: copper containing small quantities of arsenic, nickel, or tin (43—76), ordinary copper (81), H.C. copper (100), 70/30 brass (140), zinc (155), and 60/40 brass (227). C. A. King.

Mechanism of inverse segregration in alloys. R. Genders (Inst. Metals, March, 1927, advance copy, 27 pp.).—Existing theories of the cause of inverse segregation are critically reviewed, and it is considered that they all fail to account satisfactorily for the phenomenon, although the interdendritic flow theory of Bauer and Arndt (B., 1922, 220 A) is regarded as a partial explanation. The evolution of gases plays an important part, and it seems probable that inverse segregation is produced by the same mechanism in all alloys. Results of a systematic exploration of different regions in bronze ingots containing 5% Sn cast by different methods are considered with respect to ingot formation. The three essential factors in inverse segregation are a temperature range between the liquidus and solidus, the presence of dissolved gases, and an initially rapid rate of cooling, decreasing before complete solidification to below the critical rate necessary to retain the alloy as a supersaturated metal–gas solution. Inverse segregation may be minimised by processes of degasification, or by inducing solubility of the gases in the solid alloy. M. Cook.

Accurate determination of copper in bronze by electrolysis. R. A. F. Hammond (Inst. Metals, March, 1927, advance copy, 3 pp.).—Details are given of modifications of a method devised by Etheridge (B., 1924, 750) for the determination of copper in bronze which yield absolute results, accurate to $\pm 0{\cdot}02\%$, and comparatively accurate to $\pm 0{\cdot}01\%$. M. Cook.

Silver-lead ore concentration. E. W. Ellis (Eng. Min. J., 1926, **122**, 815—816).—Table concentration yielded a larger recovery of lead than of silver; the loss of silver was caused by the presence of tetrahedrite. Fine grinding, and possibly flotation, of both middlings and tailings is indicated. Chemical Abstracts.

Analysis of cassiterite, SnO_2. P. Wenger and (Mlle.) E. Rogovine (Helv. Chim. Acta, 1927, **10**, 244—245).—The cassiterite is rendered soluble by fusion with sodium peroxide. Permanganates and manganates are precipitated as manganese dioxide by warming the solution with hydrogen peroxide, and the

solution is filtered. Silica is separated from the filtrate in the usual manner, and the tin is precipitated as sulphide and weighed as dioxide. The precipitate is dissolved in hydrochloric acid and copper precipitated as sulphide. Iron, in the ferric state, is separated from any zinc, nickel, and calcium by the barium carbonate method, and these metals are then separated and determined in the usual way. R. Cuthill.

Micro-determination of small quantities of platinum in ores and minerals. G. Lunde (Mikrochem., 1927, **5**, 16—20).—1 g. of the finely-divided ore is fused with 2 g. of lead acetate, 2 g. of potassium sodium carbonate, and 2 g. of powdered borax glass in a porcelain crucible over a blast burner. The resulting lead button is scorified with borax in an unglazed porcelain crucible in a slow stream of oxygen, and finally cupelled in a similar crucible without borax. The bead of precious metal which still contains lead is heated with a blowpipe in a borax bead suspended on a quartz fibre until all lead and silver are expelled. The diameter of the resulting metallic bead is a measure of its weight. Further work is in progress to separate interfering elements, *e.g.*, the other platinum metals.

A. R. Powell.

Aluminium from Oersted to Arvida. J. D. Edwards (Trans. Amer. Electrochem. Soc., 1927, **51**, 59—62).—Historical.

Dissolution of lead by water. Farine.—See XXIII.

Patents.

Manufacture of metals and alloys in the electric furnace. D. Croese (E.P. 266,658, 27.8.25).—Finely-divided ore, *e.g.*, iron sand, or other mineral substance passes under the action of gravity through a furnace comprising a series of electrode chambers in each of which a baffle or other device is provided in order to maintain the substance under treatment in contact with the electric arcs for sufficient time. The lowest of the chambers communicates with a heated crucible in which molten metal separates from other substances produced in the electrode chambers.

J. S. G. Thomas.

Electric smelting [of zinc]. F. Tharaldsen (U.S.P. 1,618,316, 22.2.27. Appl., 1.10.23. Conv., 23.9.22).—A resistor consisting of a bath comprising an acid slag containing 15—25% Fe and 45—60% SiO_2 is used in the process of electrically smelting zinc.

M. E. Nottage.

Manufacture of metals and objects therefrom. J. Merle (E.P. 265,243, 29.7.25).—Metals or alloys are fed rapidly at temperatures well above their m.p. into rotary moulds and subjected during casting to an intense regulated centrifugal pressure. M. E. Nottage.

Cupola furnace. J. Dechesne (E.P. 265,827, 14.5.26).—The fore-hearth of a cupola furnace is adapted to be agitated mechanically; *e.g.*, the side nearest to the cupola may be mounted on a pivot, the other side being alternately raised and suddenly lowered by means of cams, the cam portions being of varying sizes.

C. A. King.

Cementation of iron and steel. R. Bellak (E.P. 260,986, 1.11.26. Conv., 4.11.25).—A carburising agent for iron and steel consists of powdered tobacco waste which may be mixed with charcoal. C. A. King.

Rendering iron and steel non-oxidisable. E. G. Caughey, Assr. to Edgewater Steel Co. (U.S.P. 1,617,616, 15.2.27. Appl., 26.1.22).—Iron is rendered non-oxidisable or resistant to oxidation at high temperatures by incorporating in the metal sufficient aluminium to form a saturated solution at the temperature at which it is desired to prevent oxidation. C. A. King.

Eliminating phosphorus from pig iron. P. C. Rushen. From Gelsenkirchener Bergwerks A.-G. (E.P. 266,033, 12.11.25).—Phosphorus is removed from pig iron by bringing the metal into contact with carbon monoxide or gases rich in carbon monoxide, and basic materials, at a temperature slightly above the m.p. of pig iron. The process is conveniently carried out by blowing carbon monoxide into a Thomas converter charged with additions of lime. C. A. King.

Manufacture of alloys of copper, nickel, and aluminium. Internat. Nickel Co., Assees. of W. A. Mudge (E.P. 250,194, 4.3.26. Conv., 31.3.25).—The physical properties of an alloy containing at least 40% Ni, up to 54% Cu, up to 17% Al, and small amounts of iron, manganese, and carbon may be materially improved by heat treatment. The alloy is first made dead-soft by quenching from 816°; subsequent heating to 316° produces no change in the physical properties; from 316° to 538° these properties show a gradual increase, reaching a maximum from 538° to 649°, above which temperature a decrease is observed. The maximum improvement is produced by extending the time of heating and decreasing the rate of cooling, and a still further slight improvement occurs as the material is aged. M. E. Nottage.

Flotation agent for use in concentrating minerals. Barrett Co., Assees. of F. G. Moses and E. J. Canavan (E.P. 243,383, 21.11.25. Conv., 22.11.24).—A flotation agent is prepared by treating acid constituents of coal tar with sulphur chloride and mixing the product with coal tar or tar oil. Or coal tar (100 pts.) may be treated directly with sulphur chloride (1—25 pts.). The sulphur compounds formed remain undecomposed for a long period. C. A. King.

Concentration of slimes by flotation. G. A. Bragg, Assr. to Metals Recovery Co. (U.S.P. 1,619,790, 1.3.27. Appl., 28.9.26).—Slimes consisting mostly of particles smaller than 200-mesh are agitated vigorously with lime to condition the sulphide minerals for a subsequent flotation operation in the presence of a non-oleaginous mineral-collecting agent. C. A. King.

Extraction of precious metals from their ores and concentrates. Mond Nickel Co., and R. H. Atkinson (E.P. 266,040, 14.11.25).—A mixture of an oxidised ore or concentrate, lead, and zinc (or zinc alloy) is heated at a temperature between the m.p. of lead and the sintering point of the ore, using one or more chlorides as a flux. The metallic beads are separated from the residue and the precious metals are recovered by a process similar to Parkes' process. The residue may be treated by magnetic means and retreated for further recovery of metallic values. C. A. King.

Soldering aluminium or its alloys. L. R. Preston (E.P. 265,733, 14.12.25).—A solder for aluminium or aluminium alloys is composed approximately of 75% Sn, 22% Zn, 2% Al, and 1% Bi. The solder is coated with a mixture of 25% of stearic acid, 25% of silver sand, and 50% of phosphor-tin (5%). C. A. King.

Production of mechanically very solid bodies of suitable shapes from carbides of metals having high fusing points, such as tungsten carbide, for tools and like articles. H. Lohmann (E.P. 246,487 and 248,336, 21 and 22.1.26. Conv., [A] 24.1.25, [B] 24.2.25). —(A) An acidic oxide, anhydride, or imperfectly reduced oxide of a highly refractory metal, *e.g.*, tungsten, is mixed with a small percentage of thorium, uranium oxide, tantalum or its oxide, or molybdenum oxide, and the mixture fused when actually in a carbon mould of the desired shape. (B) For the production of tools of tungsten carbide, raw unfused tungsten is mixed with thorium carbide (5—10%) and fused, as distinct from sintering in a carbon mould. The addition of 3—5% of molybdenum aids the homogeneity of the product.
C. A. King.

Magnetic alloys and their application in the manufacture of telegraphic and telephonic cables. W. S. Smith and H. J. Garnett (E.P. 266,066, 20.11.25). —Alloys suitable for the manufacture of wire or tape for use in telegraphic or telephonic cables (see B., 1925, 76) contain 75—76% Ni, 16—18% Fe, 5% Cu, a small quantity of manganese, and 2% of an element to increase the electrical resistance, *e.g.*, tungsten, chromium, silicon, titanium, or molybdenum.
C. A. King.

Pulverising and separating minerals (E.P. 265,841).—See I.

Compact coke (G.P. 435,305).—See II.

XI.—ELECTROTECHNICS.

Use of electric furnaces at Niagara Falls, 1902 to 1926. F. A. J. FitzGerald (Trans. Amer. Electrochem. Soc., 1927, **51**, 47—50).

Electrometric determination of ash in sugar. Šandera.—See XVII.

Patents.

Insulating high-tension electric currents. O. Y. Imray. From Aktien-Ges. für Anilin-Fabr. (E.P. 244,068, 12.11.25).—The product obtained by the removal of olefines from the extract resulting from the treatment of suitable mineral oils, coal tar, etc. with liquid sulphur dioxide, as in the Edeleanu process, is used as insulating material. J. S. G. Thomas.

Electric [arc] furnace. British Thomson-Houston Co., Ltd., Assees. of V. J. Chapman (E.P. 249,880, 25.3.26. Conv., 26.3.25).—An electric arc furnace comprising a furnace chamber and a refractory crucible is provided with a bottom electrode arranged between the wall of the crucible and the wall of the chamber, and having its lower end completely enclosed in a wall of the furnace. Electrical connexion with this electrode is made by a member of relatively high electrical conductivity which is maintained in good electrical engagement with the electrode at a point within the furnace, although the respective coefficients of expansion of the different parts may be unequal. The part of this conducting member projecting outside the furnace forms a terminal, and is provided with cooling means.
J. S. G. Thomas.

Electric furnace for melting or producing glass, water-glass, Portland cement, aluminous cement, etc. C. E. Cornelius (E.P. 249,554, 19.3.26. Conv., 23.3.25). — The substance to be melted or produced forms the furnace resistance, and is heated between or in contact with metallic electrodes which may be corrugated so as to be of large area.
J. S. G. Thomas.

Filling element. F. Ehrhardt (G.P. 435,028, 7.10.24).—Electrodes wrapped in capillary material are saturated with a reversible colloidal solution and dried. The device ensures uniform distribution and adequate rigidity of the electrolyte employed.
J. S. G. Thomas.

[Electrolytic] means for indicating the presence of saline, alkaline, and acid impurities in water and other fluids. W. Crockatt & Sons, Ltd., and W. C. Crockatt (E.P. 266,047, 16.11.25).—In conjunction with a conductivity cell containing the fluid, and provided with electrodes connected to a source of current, means are provided for indicating a change of electrical conductivity of the fluid, and a variable resistance or potentiometer is inserted in the external circuit in order to vary the current in the circuit. The effect of variation of temperature upon the indication is automatically eliminated. J. S. G. Thomas.

Treatment of cellulose (F.P. 610,417).—See V.

Phosphoric acid and phosphates (G.P. 434,985).—See VII.

Arc shield (U.S.P. 1,617,515).—See VIII.

Magnetic alloys for cables (E.P. 266,066).—See X.

XII.—FATS; OILS; WAXES.

Opaque drying of tung oil. O. Merz (Chem. Umschau, 1927, **34**, 45—49).—The author observes the behaviour, particularly changes in appearance, of films of raw and "treated" tung oils (boiled oil, stand oil, etc.) dried in desiccators and in the open under various conditions—different sources of light, presence or absence of air currents, etc. The theory that ultraviolet light is responsible for the frosting, webbing, and similar phenomena observed in the drying of tung oil films is untenable, since clear films are obtained under the pure ultra-violet light of the analytical quartz mercury-vapour lamp. The experimental conditions chosen establish temperature fluctuation as the deciding factor. Such fluctuation and not any action ascribable to the gas mixture is the explanation of the "coal-gas test." Tung oil films were found to dry without frosting in the desiccator in a constant-temperature stream of the products of combustion of coal gas. S. S. Woolf.

Detection of whale oil. J. Davidsohn (Chem. Umschau, 1927, **34**, 49—50).—The method suggested by Tsujimoto (B., 1926, 986) for detection of whale oil by means of the turbidity and precipitates given by its

freed fatty acids with Wijs reagent under specified conditions was not corroborated on examination. The octabromide test remains the only reliable one for normal whale oil, and a qualitative modification is described. Bromides are prepared by shaking the fatty acids isolated from the oil under test with Halphen's reagent (4 vols. of nitrobenzene, 1 vol. of bromine, and 28 vols. of glacial acetic acid). Octa- and deca-bromides characteristic of whale oil are insoluble in benzene.

S. S. WOOLF.

Odoriferous oil and linoleic tetrabromides from Philippine lumbang oil. S. SANTIAGO and A. P. WEST (Philippine J. Sci., 1927, **32**, 41—52; cf. West and Montes, B., 1921, 896 A).—By hydrolysis of the glycerides and conversion of the potassium soaps into zinc soaps, removal of these, and extraction of the residue with ether, a colourless oil, b.p. 80—95°, soluble in ether and in water, containing aldehydes and unsaturated compounds, and having an intense lumbang odour, was obtained; the yield was about 2%. The ethereal solution of the free mixed acids was brominated at $-10°$, the insoluble linolenic hexabromide removed, the solution evaporated to dryness, and the residue washed with light petroleum to remove oleic dibromide. By fractional crystallisation, first from ethyl and then from methyl alcohol, four tetrabromides were obtained; α, m.p. 112·3—114·3°; β, m.p. 59—60°; γ, liquid; and δ, m.p. 57—58°. It is suggested that lumbang oil contains the four possible geometrically isomeric linoleic acids.

S. I. LEVY.

Heats of combustion of some oils from marine animals. H. MARCELET (Compt. rend., 1927, **184**, 604—605).—The heats of combustion and temperatures of inflammability and combustion of a number of oils from marine animals, and of squalene, have been determined with the object of using the oils in semi-Diesel engines. The highest calorific powers recorded are of the same order as those of the petrols.

J. GRANT.

Determination of the iodine value [of oils] in aqueous emulsions. J. FIALKOV (Z. anal. Chem., 1927, **70**, 227—229).—The following modification of the method of Margosches (B., 1924, 639) gives good results with oils, but not quite satisfactory results with fats: 0·1—0·15 g. of oil is mixed with half its weight of powdered gum arabic and one or two drops of water to a smooth emulsion to which are added 5—10 c.c. of water and 20 c.c. of $0 \cdot 2N$-iodine solution. After diluting to 200 c.c. the liquid is shaken vigorously and titrated after 5 min. with thiosulphate as usual. A. R. POWELL.

Analysis of alkaloids. WATKINS and PALKIN.—See XX.

PATENTS.

Preparation of a non-gelatinising tung-oil product. G. HALL (G.P. 434,202, 12.10.21).—The gel obtained by heating tung oil alone or in the presence of catalysts at a temperature below 200° is reliquefied by further heating at 250—300° to form a varnish base that may be combined with other oils, resins, etc.

S. S. WOOLF.

Manufacture of a drying and polymerising oil. J. J. KESSLER (U.S.P. 1,616,321, 1.2.27. Appl., 9.5.24).—The coal-tar fraction, b.p. 150—250°, is heated with an excess of formaldehyde or other aldehyde in presence of a trace of ammonia or sodium hydroxide at 150°. The oil obtained dries on exposure to air, and slowly polymerises at 150°. T. S. WHEELER.

Manufacture of highly viscous lubricating oils. G. SCHICHT A.-G. (E.P. 250,947, 14.4.26. Conv., 14.4.25).—A mineral oil–fatty oil blend suitable for use as a lubricant, particularly at high temperatures, is obtained by polymerising a drying oil in a current of non-oxidising gas at about 300° until the multiply unsaturated fatty acids are for the most part or completely saturated, drying power being lost accordingly. From ⅓ to 1 pt. of mineral oil is added to 1 pt. of drying oil, either at the outset or during the heating operation, the latter being discontinued before the thickened drying oil becomes insoluble in mineral oil. S. S. WOOLF.

Production of vitaminised oils. A. W. OWE (E.P. 266,139, 2.3.26).—To obviate the reduction in vitamin content unavoidable in attempts to remove the objectionable flavour and smell from vitamin concentrates before introducing them into edible oils, the unrefined vitamin concentrate is dissolved in the oil, and the resulting solution is subjected to a mild refining treatment, depending on the nature of the concentrate, in the absence of air and at low temperatures.

S. S. WOOLF.

Refining oils. F. J. RESINES, Assr. to BY-PRODUCTS RECOVERY CO. (U.S.P. 1,619,486, 1.3.27. Appl., 28.9.23).—Vegetable oil is agitated, neutralised with caustic alkali solution, and immediately separated by centrifugal action. The neutral oil is again similarly treated to decolorise it. H. ROYAL-DAWSON.

Manufacture of soap. A. C. NESFIELD and L. F. GOODRICKE (E.P. 266,435, 23.11.25).—Optimum proportions of the saponifiable material and the alkaline reagent, both heated slightly below 100°, are brought into intimate contact in a described emulsifying vessel, fitted with baffles and other suitable devices. The emulsifier discharges the material on to the surface of a spreading disc (rotated if desirable) in a boiling vat provided with coils to which superheated steam at 315° is admitted. Before discharge into a secondary vessel, in which glycerin recovery and admixture of colouring matter, scents, etc. take place, the boiling liquid can be circulated through the emulsifier as many times as is found necessary according to the nature of the substance under treatment. S. S. WOOLF.

Manufacture of soap [flakes]. E. FLAMMER and C. KELBER (E.P. 265,714, 23.11.25).—The water content of soap made from a mixture of saturated fatty acids and unsaturated fatty acids of the erucic or ricinoleic type is reduced to 8—14%, and perborates or other compounds containing active oxygen are incorporated. Hydrocarbons, *e.g.*, hexadecane, or their hydroxy-compounds, *e.g.*, slightly oxidised paraffin wax, cetyl alcohol, cetyl palmitate, may be added at this stage. Such mixtures are not brittle, and can be converted satisfactorily into thin flakes, a suitable rolling and cutting apparatus being described. A soap mixture consisting of 17% of potassium soaps and 83% of sodium soaps is preferred. S. S. WOOLF.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Patents.

Producing light-resistant lithopone. J. E. Booge, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,617,311, 8.2.27. Appl., 22.10.23. Renewed 30.4.26. Cf. U.S.P. 1,535,760; B., 1925, 514).—Crude lithopone of low chloride content, which has been calcined in presence of a small quantity of oxygen, is quenched with water, acidified with sulphuric acid, washed with water and with sodium carbonate, blued, dried, and ground. It is resistant to light, and has a good colour.

T. S. Wheeler.

Increasing the resistance of lithopone to sunlight. H. G. Schanche, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,619,376, 1.3.27. Appl., 5.9.23).—Crude lithopone is calcined and quenched, and after wet-grinding the product it is treated with an acid solution. H. Royal-Dawson.

Ingredient for printing ink. J. H. Caughlan (E.P. 265,787, 22.2.26).—A mixture of aluminium resinate and gelatinous aluminium hydroxide, prepared by precipitating a boiling sodium resinate solution with aluminium sulphate, is washed, filtered, and ground as a wet pulp with lithographic varnish. The product is used in printing inks. S. S. Woolf.

Dyeing inks. I. G. Farbenind. A.-G. (F.P. 608,903, 2.1.26. Conv., 3.1. and 13.1.25).—Formaldehyde and/or an alkali, or substances producing them, are added to solutions of substantive dyestuffs. S. S. Woolf.

Production of solutions for lacquering, impregnating, coating, doping, etc. of fabrics, fibrous and solid materials, etc. A. Eichengrün (E.P. 243,031, 17.11.25. Conv., 17.11.24).—Non-inflammable or difficultly inflammable solutions are obtained by dissolving acetone-soluble cellulose acetate (or a mixture of acetone- and chloroform-soluble cellulose acetates in which the acetone-soluble variety preponderates) in a mixture of methylene chloride (a non-solvent for cellulose acetate) with small amounts of inflammable solvents, *e.g.*, alcohol, acetone, etc., in which mixture cellulose acetate is soluble. Relatively concentrated solutions and films of high lustre are produced by this method. S. S. Woolf.

[Nitrocellulose] varnishing preparation. K. Takemura and K. Oiwa (E.P. 266,214, 12.8.26).—A varnish suitable for the protection of ships' bottoms, and having good insulating properties, consists of 15 pts. of nitrocellulose, 35 pts. of amyl acetate, and 10 pts. each of ether and acetone (solvents), 20 pts. of alcohol or benzol (diluents), 5 pts. of dibutyl tartrate (plasticiser), 5 pts. of pulverised bamboo (filler), and 3 pts. of potassium ferrocyanide (anti-corrosive agent). S. S. Woolf.

Preparation of dyed lacquers. I. G. Farbenind. A.-G. (Swiss P. 115,116, 18.2.25. Conv., 1.3.24).—Aqueous suspensions of dyestuffs are mixed with cellulose ester solutions compatible with the water so introduced. A typical solvent mixture is acetone, butyl alcohol, *cyclo*hexanol, *cyclo*hexyl acetate, and butyl acetate.

S. S. Woolf.

Oxidation of rosin (colophony) by air or oxygen. A. Riebeck'sche Montanwerke A.-G. (G.P. 434,419, 12.3.22).—Rosin in solution in low-boiling solvents, *e.g.*, glacial acetic acid, is oxidised in the presence of catalysts, *e.g.*, sodium nitrate, to yield shellac substitutes.

S. S. Woolf.

Manufacture of artificial resins. British Cyanides Co., Ltd., and E. C. Rossiter (E.P. 266,028, 5.11.25).—A mixture of carbamide and thiocarbamide is condensed with formaldehyde, or the separate condensation products are blended at an intermediate stage in their preparation. The combined resin retains the solubility of the thiocarbamide resins, but "cures" more rapidly. If 25% or less of the thiocarbamide be replaced by carbamide, clear resin syrups are obtained. Above this figure the resins obtained are turbid, but "cure" rapidly, and may be clarified partially by the addition of ammonia. S. S. Woolf.

Preparation of linoxyn-like substances. Consortium für Elektrochem. Ind., G.m.b.H., Assees. of H. Deutsch and W. O. Herrmann (G.P. 434,318, 19.6.24).—Aldehyde-resins are incorporated with hydroxy-acids or substances containing them, subjected to a preliminary or continued oxidation. Further additions are made, *e.g.*, resins, waxes, tung oil, hydrogenated phenols or hydrogenated naphthalene, etc. The products are used in the manufacture of linoleum, artificial leather, ivory-substitutes, etc.

S. S. Woolf.

Composition of material [resin]. O. A. Cherry, Assr. to Cutler-Hammer Manuf. Co. (U.S.P. 1,616,741, 8.2.27. Appl., 18.6.23).—Sulphur chloride (200 pts.), sulphur or selenium (50—150 pts.), and phenol (100 pts.) react when mixed to form a synthetic resin. In place of phenol, naphthalene or anthracene can be employed. T. S. Wheeler.

Chromo-lithographic transfers. H. Courmont (E.P. 265,651, 9.10.25).

Waterproofing fibre (U.S.P. 1,616,062).—See V.

Decorated paper (E.P. 265,334).—See V.

Light filters (E.P. 265,455).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Patents.

Vulcanisation of rubber. I. G. Farbenind. A.-G., Assees. of W. Kropp (U.S.P. 1,616,378, 1.2.27. Appl., 5.3.26. Conv., 6.8.25).—The products obtained by the condensation of $\alpha\beta$-dialkylacraldehydes, *e.g.*, α-ethyl-β-propylacraldehyde, with aniline or other aromatic amine, are of value as accelerators for the vulcanisation of rubber. They are oily liquids, and can be obtained by heating the components together, with or without addition of a solvent, *e.g.*, benzene, and a condensing agent, *e.g.*, zinc chloride. T. S. Wheeler.

Vulcanising rubber. M. L. Weiss, Assr. to Dovan Chemical Corp. (U.S.P. 1,616,936, 8.2.27. Appl., 29.7.25. Renewed 5.11.26; cf. U.S.P. 1,411,231 and 1,546,876; B., 1922, 383 a; 1925, 1000).—Compounds of the type $NH:C(NHR)(NR_2)$, where R = aryl, *e.g.*, *as*-triphenylguanidine, are of value as accelerators of the vulcanisation of rubber. About 1% of the weight of the rubber is required.

T. S. Wheeler.

Method of vulcanising rubber. L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,616,994, 8.2.27. Appl., 8.11.24).—Piperazine or a derivative, *e.g.*, piperazine stearate, is used as an accelerator.

T. S. WHEELER.

Vulcanising rubber. T. W. MILLER, Assr. to FAULTLESS RUBBER Co. (U.S.P. 1,617,411, 15.2.27. Appl., 12.5.23).—Rubber (100 pts.) is dusted on the surface with a mixture of a metallic oxide (10 pts.), sulphur (3 pts.), and an organic accelerator (2 pts.), and heated. Vulcanisation occurs at a comparatively low temperature.

T. S. WHEELER.

Manufacture of goods from india-rubber. A. FRASER (E.P. 266,041, 14.11.25).—Receptacles such as battery boxes, with a hard vulcanite interior and a soft elastic rubber exterior, are formed from a rubber mixture by differential vulcanisation, *e.g.*, by applying heat only to the portion of the mould in contact with the cavity, thereby ensuring the necessary temperature gradient in the wall.

D. F. TWISS.

Process and apparatus for concentration of [rubber] latex. K.D.P., LTD. (E.P. 255,014, 13.11.25. Conv., 9.7.25. Addn. to E.P. 244,727; B., 1927, 52).—Rubber latex is concentrated in an externally heated, rotating drum; a loose roller resting in the lowest part of the drum serves as a spreading device. The rate of evaporation may be controlled by altering the volume and temperature of the air passing through the drum.

D. F. TWISS.

Production of aqueous rubber dispersion. W. B. PRATT, Assr. to RESEARCH, INC. (U.S.P. 1,621,468, 15.3.27. Appl., 2.7.23).—See E.P. 217,612; B., 1924, 757.

XV.—LEATHER; GLUE.

Tannin–gelatin reaction. I. P. BAUGHMAN (J. Physical Chem., 1927, **31**, 448—458; cf. Thomas and Frieden; A., 1923, ii, 664).—The determination of tannin in small amounts, the tannin–gelatin reaction near the maximum flocculation point, and the effects of various electrolytes and of the age of the gelatin on this reaction, have been studied. 1 c.c. of a saturated solution of ferrous ammonium sulphate is added both to 10 c.c. of the tannin solution containing not more than 0·0002 g./c.c., and to 10 c.c. of a standard solution of pure tannin (Kahlbaum), and the two solutions are compared colorimetrically. The method is sensitive to $\pm$ 0·000005 g./c.c. In the tannin-gelatin reaction maximum flocculation occurs with 0·5% of tannin and gelatin solutions, but beyond this point the protective action of an excess of gelatin on the tannin is marked. Gelatin can be completely precipitated by tannin, but some tannin remained in solution in all the series used. Sodium chloride (1%) reduces the amount of tannin remaining in the filtrate, but 1—4% solutions of sodium sulphate or of magnesium, aluminium, and mercuric chlorides increase this amount. In agreement with Thomas and Frieden (*loc. cit.*), the age of a sterilised gelatin solution has no effect on the tannin-gelatin reaction.

L. S. THEOBALD.

PATENTS.

Manufacture of chamois and like leather. A. KEMMLER (E.P. 266,622, 13.9.26).—Skins to be tanned are prepared in the usual way and dressed in a fulling mill with egg oil to which 0·5—2% of 40% aqueous formaldehyde is added (according to the thickness of the leather). The customary drying and softening operations are then employed.

S. S. WOOLF.

Production of mineral-tanned leather. F. W. WEBER (U.S.P. 1,616,400, 1.2.27. Appl., 19.3.25. Cf. U.S.P. 1,221,929; B., 1917, 605).—Dry pickled hides are soaked in sodium chloride solution, to which is then added a solution of the basic acetates of the rare-earth metals. The hides are dried after 3—5 hrs., acetic acid being evolved, and oxides of the metals deposited on the hides, which are then returned to the bath containing the basic acetates. To this solution is now added a colloidal solution of ferric chloride which has been neutralised by addition of carbonates of the rare-earth metals. Tanning is complete in 2 hrs. The hides, when dried and treated with a neutral soap solution at 40—50°, yield a strong waterproof leather. In place of ferric chloride, chromium alum and aluminium sulphate can be employed.

T. S. WHEELER.

Manufacture of horn-like materials from albuminous substances. DEUTSCHE KUNSTHORN G.M.B.H. (E.P. 247,955 and 261,009, [A], 16.2.26. Conv., 21.2.25. [B], 4.11.26. Conv., 4.11.25. Cf. B., 1926, 840).—(A) Paraformaldehyde that has been previously treated with acids, *e.g.*, sulphuric acid, formic acid, etc., is used as hardening agent in the preparation of artificial horn from albuminous substances, *e.g.*, casein, the acid treatment retarding the hardening action of the formaldehyde sufficiently to allow plastification and moulding to take place. The moulded plastic mass is then dried and hardens. (B) The moulded plastic masses containing hardening agents are preferably allowed to remain quiescent in closed vessels for a varying period before the drying and hardening operations are undertaken.

S. S. WOOLF.

XVI.—AGRICULTURE.

Basis of microbiological experiments in soils. A. RIPPEL (Z. Pflanz. Düng., 1927, **A**, **8**, 268—276).—The lack of co-ordination between field observations and the results of laboratory culture experiments with soil organisms is discussed from the point of view of the difference in the environment of the organisms necessitated by the present methods of microbiological technique.

A. G. POLLARD.

Microbiology of forest soil. R. BOKOR (Biochem. Z., 1927, **181**, 302—304).—In light, well-aerated, sandy soil, aerobic bacteria are predominant, the production of carbon dioxide and putrefactive changes are most intense, the amounts of humus and organic matter least, and the number of cellulose-attacking bacteria is proportional to the production of carbon dioxide by the soil. In an alder swamp, however, the number of bacteria is large, but the production of carbon dioxide is small owing to the high percentage (56%) of water which enables anaerobic bacteria to predominate.

P. W. CLUTTERBUCK.

Buffer capacity in acid soils. H. KAPPEN (Z. Pflanz. Düng., 1927, **A**, **8**, 277—288).—The term "buffer capacity" may cover a number of chemical and physical

processes, *e.g.*, neutralisation, dissociation, ampholytic action, adsorption, etc. Its value from this general standpoint is discussed. The measurement of "buffer area" (Jensen) is considered a valuable figure in the characterisation of soils. Measurements based upon p_H values are not always expressive of the true condition of soils, and methods involving these measurements (*e.g.*, Arrhenius) become of value only when actual changes in hydrogen-ion concentration following the addition of acid or alkali are compared.

A. G. POLLARD.

Physiological significance of nutrient extracts in the determination of the manurial requirement of soils by chemical means. E. BLANCK and F. SCHEFFER (Z. Pflanz. Düng., 1927, **A**, **8**, 298—302).—Comparison is made of measurements of the availability of potassium and phosphate in soils by the acid extraction method and that of Neubauer. Values for phosphate showed fair agreement, but discrepancies are apparent in comparing values for available potash. The nature and concentration of acid used in preparing extracts have a much greater effect on the solubility of potash than of phosphates. Owing to the different nature of the compounds of potash and of phosphate in soils it is not to be anticipated that any one solvent will give a satisfactory measure of the availability of both. A. G. POLLARD.

Pot experiments with various fertilisers. E. BLANCK, F. GIESECKE, and F. SCHEFFER (Z. Pflanz. Düng., 1927, **B**, **6**, 49—67).—The results of pot experiments with some new fertilisers are given. "Leunaphos," prepared by mixing ammonium sulphate with diammonium phosphate, proved equally efficient with other soluble nitrogenous and phosphatic fertilisers. "Biophosphat," a preparation containing mineral phosphate and estuarine alluvial clay containing a high proportion of colloidal silicic acid, proved considerably less efficient than ordinary phosphatic fertilisers. "Schlick-Kalkstickstoff," a similar preparation, but containing nitrogen, also proved unsatisfactory. "Asahi-Promoloid," a colloidal silicic acid preparation, showed some effect in pot experiments, but no effect in field experiments.

G. W. ROBINSON.

Determination of the fertiliser requirements of soils [by the Neubauer seedling method]. H. WIESSMANN (Z. Pflanz. Düng., 1927, **B**, **6**, 68—82).—In experiments on the utilisation of phosphoric acid by seedlings using the Neubauer method, utilisation was complete only with the smallest dressings. The seedling method cannot therefore be used to determine exactly the content of available phosphoric acid in soil. The method gives satisfactory results for the available potassium content of soils. G. W. ROBINSON.

Manuring experiments on hops, 1925. A. H. BURGESS (J. Inst. Brew., 1927, **33**, 138—140).—Two series of plots at Chilham and Horsmonden each comprise six different manurial treatments. Taking the average of the crops for the years 1922—1925, the effects of the various treatments at Chilham bear the following relationships:—Complete manure (mineral) 100·0; complete manure (including dung) 99·5; without phosphate 94·0; without nitrogen 60·2; without potash 51·6; unmanured 38·8. The corresponding figures for Horsmonden are:—Complete manure (mineral) 100·0; without phosphate 96·7; without potash 95·0; complete manure (including dung) 81·0; unmanured 59·5; without nitrogen 57·9. No conclusion can be drawn at present with regard to the effect of manuring on the preservative value of the hops. C. RANKEN.

Influence of boron on the growth of the soya bean plant. G. H. COLLINGS (Soil Sci., 1927, **23**, 83—105).—Experiments are described on the effect of boron on the germination and growth of soya beans in water, sand, and soil cultures. In water culture, 0·4—0·5 mg. of boron per litre of nutrient solution is toxic to soya beans in the seedling stage, and concentrations as low as 0·1—0·2 mg. per litre cause characteristic injury to the leaves of older plants. The toxicity is practically the same whether the boron is present as boric acid or as potassium or sodium borate. Boron is not necessary for the growth of seedling soya beans, nor for the production of mature plants. In sand cultures, germination is delayed or inhibited by the presence of boron. In sand and soil cultures, injury to the leaves and a reduction of dry weight of the plants was caused by the use of sodium borate or its equivalent at the rate of more than 1 lb. per acre. No appreciable stimulation due to boron was noted with soya beans grown in sand or soil, or with seedlings in water culture, but with plants grown to maturity in water culture, boric acid, and to a less degree sodium borate, exerted a stimulating influence, which was greatest at 2·5 mg. of boron per litre of solution. C. T. GIMINGHAM.

Organic matter changes in two soil zones, as influenced by difference in form, fineness, and amount of calcium and magnesium compounds. W. H. MACINTIRE, W. M. SHAW, and E. M. CRAWFORD (Soil Sci., 1927, **23**, 107—117).—Experiments are reported on the changes of organic matter in an originally uniform soil, due to addition of calcium hydroxide, mixtures of calcium and magnesium oxides, limestone, or dolomite, the last two at varying degrees of fineness. In one series the materials were incorporated with the surface half or zone, and in another, with the lower half (subsurface zone), the soil being exposed to natural leaching in lysimeters and kept uncropped and unstirred. In general, oxidation of organic matter was accelerated by the additions, the effects in the two zones being comparable. Conditions in the subsurface zone, even without additions, were, however, more conducive to oxidation than in the surface zone. No positive differences were attributable to variation in the fineness of the limestone. C. T. GIMINGHAM.

Contraction and expansion of soils when wetted with water. G. J. BOUYOUCOS (Soil Sci., 1927, **23**, 119—126).—When absolutely dry soils are wetted with water, the original volume of soil and water decreases. The amount of contraction varies with different soils, and bears a close relationship to the content of organic matter and to the heat of wetting of the soils. It is also related to the colloidal content, but less closely and consistently. The contraction in volume is attributed principally to condensation of some of the water on the surface of the colloids. The determinations were made in a special dilatometer, the method involving the

temporary exclusion of water from the dry soil by the use of carbon tetrachloride. C. T. GIMINGHAM.

Absorption of moisture by soils. J. N. SEN and B. M. AMIN (Mem. Dept. Agric. India, 1926, **8**, 235—253).—In nine out of eleven soils tested, the amounts of water absorbed from an atmosphere in equilibrium with a 25% aqueous solution of glycerin were dependent on the percentages of clay. The two exceptions were unusual types of soil, and it is suggested that the determination of the absorbing capacity gives useful indications as to the type of colloids present in the soil and its behaviour under cultivation. Determinations at 20—60° showed that the absorbing capacity decreases as the temperature rises. C. T. GIMINGHAM.

Colloidal behaviour of soils and soil fertility. III. Cation replacement and saturation of soil with calcium. J. S. JOFFE and H. C. MCLEAN (Soil Sci., 1927, **23**, 127—135. Cf. B., 1925, 858; 1926, 457). —The relation of the various exchangeable cations present in the soil complex and the methods used in determining base-exchange capacity and degree of unsaturation of soils are discussed. Experimental data on the relation between "lime requirement" and degree of unsaturation are given, and it is suggested that a determination by the replacement method gives results from which the "lime requirement" can be obtained. The possibility of subsoiling for the purpose of preserving bases that are being leached out from the surface soils is considered. C. T. GIMINGHAM.

Factors influencing efficiency of different forms of nitrogen as related to soil type and cropping system in the Atlantic coastal plain region. I. A. M. SMITH (Soil Sci., 1927, **23**, 137—164).—The influence of temperature and water content of a sandy loam soil on the rate of nitrification of various nitrogenous materials, and the leaching of nitrates from the soil, as influenced by these materials, have been studied. The data have a bearing on the choice of a fertiliser which will yield sufficient nitrates for rapid early growth, but will not give a large accumulation to be lost by drainage. C. T. GIMINGHAM.

Nitrifying power of some Philippine soils. M. M. ALICANTE (Philippine J. Sci., 1927, **32**, 1—27).—The nitrifying powers of over 100 samples from six different provinces, to which ammonium sulphate, alone and with a slight excess over the theoretical quantities of calcium carbonate, and copra cake, alone and with slight excess of calcium carbonate, had been added, are tabulated, and are found to be closely proportional to the productivities. Soils devoted to sugar cane converted their original nitrogen more slowly than did those devoted to rice, hemp, tobacco, coconut, and citrus; soils containing high proportions of clay converted both original and added nitrogen very slowly. Addition of calcium carbonate generally accelerated nitrification, except in one case when applied with copra cake. Ammonium sulphate was generally converted more rapidly than the cake. The nitrifying power of Philippine soils is on the average lower than that of Hawaiian but higher than that of United States soils. S. I. LEVY.

Nature of soil acidity. H. J. PAGE (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, **A**, 232—244).—The views of a number of workers are discussed and shown to be capable of explanation on a common basis, if the conception is admitted that the absorbing complex of the soil is an insoluble colloidal acid (or acidoid) associated with surface-active basic and hydrogen cations. The existence of replaceable aluminium ions in the complex and the possibility of hydroxyl-ion adsorption are debatable points. A. G. POLLARD.

Soil acidity. S. D. CONNER (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, **A**, 18—21).—The nature of the acidity of a number of Indiana soils can be explained adequately by Kappen's theories. Hydrolytic and exchange acidity is ascribed mainly to the presence of acid silicates resulting from the leaching out of bases by solutions of weak organic acids and carbon dioxide. Both hydrogen and aluminium ions exist in acid soils. The toxicity of these varies with different plants. It is seldom profitable or necessary to neutralise all of the hydrolytic acidity by liming. In acid soils containing much soluble aluminium, treatment with phosphates is even more valuable than liming. A. G. POLLARD.

Relation between soil acidity and the physiologically acid reaction of some fertiliser salts. H. KAPPEN (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, **A**, 245—248).—Changes in soil acidity brought about by the use of physiologically acid or basic fertilisers were examined by the Neubauer method. Except in soils heavily denuded of replaceable bases the main cause of plant toxicity in acid soils lies within the soil itself, and is not markedly affected by the physiological reaction of added fertilisers. A. G. POLLARD.

Value of iron in zeolitic silicates in regard to soil reaction. L. SMOLIK (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, **A**, 25—28).—Aluminium in zeolitic silicates may be partially replaced by iron. In soils containing such substituted zeolites it is impossible to judge the reaction by determinations of the molecular structure of the silicate. Where the molecular ratio $SiO_2 : Al_2O_3$: other bases is $3 + : 1 : < 1$, the soil may be neutral or alkaline if more than 0·6 mol. of iron replaces aluminium. A. G. POLLARD.

Determinations of p_H of soil by the Biilmann quinhydrone method. D. J. HISSINK and J. VAN DER SPEK (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, **A**, 29—40).—In using the quinhydrone electrode for determining p_H values of soil suspensions, the following details of technique were found advantageous. A period of 30 min. is allowed between the immersion of the electrode and the measurement of the potential difference. For the complete peptisation of the clay in air-dried samples of soil, the suspension should be kept about 20 hrs. Soil suspensions are preferable to centrifuged extracts for the determination. Maximum p_H figures are recorded when a suspension of 1 pt. of soil in 4 pts. of water is used. It is important to adhere strictly to a particular soil : water ratio in comparative work. A. G. POLLARD.

Action of non-diffusible ions in soil phenomena. N. M. COMBER (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, **A**, 43—45).—The presence of non-diffusible (or slowly diffusible) ions in the soil solution and of continuous membranes formed by colloidal matter

forms the basis of the conception that ionic concentration in the soil solution varies in different local areas of the soil. A. G. Pollard.

Reaction of Finland soils. W. Brenner (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, A, 48—54).—Acidity in Finnish soils is influenced by the water level in the subsoil, which determines the level at which acid aluminium salts are concentrated. The formation of humus which has a p_H value of 3·5—6·5 also tends to render surface soils acid, irrespective of the influence of the subsoil. In a general classification of soils, buffer values should be expressed as alterations in p_H values resulting from the addition of definite amounts of acid or alkali. For comparative work, the best measure is the " reaction amplitude," viz., the sum of p_H changes following the addition of a definite amount of acid and an equivalent amount of alkali. A. G. Pollard.

Determination of exchangeable cations, degree of saturation, and relative acidity of soils. A. A. J. von Sigmond (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, A, 55—71).—Comparison is made of the methods of Hissink, Gedroiz, and Kelley for determining the degree of saturation of exchangeable bases in soils. Simple determination of p_H values of soils can give no reliable information as to their lime requirement. The value $(T - S)$ [Hissink] is too high, and better results are obtained by electrometric methods. The use of sodium chloride solutions in preparing soil extracts for this purpose is unreliable for soils containing considerable amounts of finely-divided calcium carbonate and sulphate and/or magnesium carbonate. p_H Values should be supplemented by measurements of buffer capacity and exchange acidity. This is particularly emphasised in the case of alkali soils.
A. G. Pollard.

Determination of the lime requirement of the soil. H. R. Christensen and S. T. Jensen (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, A, 94—115).—Acid soils were treated in the field with varying amounts of lime, and curves obtained showing the relationship of lime added to the p_H value produced. Similar curves were obtained in the laboratory by adding successive amounts of calcium carbonate to soil suspensions. In general, the lime required to produce p_H 6·8—7·2 in the field was three times that in laboratory tests. Sandy soils required more lime to produce a specific change in p_H than did loams. The reverse is the case for the mineral acidity determined by the Daikuhara method. The acidity of sandy soils depends more on humic matter than in the case of loams. The Daikuhara method for lime requirement usually indicated too low a figure. A. G. Pollard.

Determination of the lime requirement of humus sandy soils. J. Hudig (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, A, 116—125).—In the examination of the effect of lime on humus soils it is considered that the basis of comparison should be the amount of humus present (loss on ignition) rather than the total amount of soil. The buffer capacity of the humus has a definite bearing on the lime requirement of the soil. Repeated field trials and pot experiments are necessary before the lime requirement of a soil can be satisfactorily determined. The " lime condition " of a humus soil is expressed as the number of g. of calcium carbonate required to neutralise 100 g. of humus. Negative values indicate lime required. Biilmann's quinhydrone electrode is the most satisfactory for p_H determinations in this work. A. G. Pollard.

Lime requirement of soils. A. Gehring (Z. Pflanz. Düng., 1927, A, 8, 257—267).—Recognised methods for determining the lime requirement of soils are discussed from the point of view of their effectiveness in soils of different types. A modification of the Hissink method is suggested. The degree of saturation is measured by the original method, but in measuring the total capacity of a soil for calcium the sample is prepared by shaking with lime water for 20 hrs. Excess lime is removed by means of carbon dioxide, and the adsorbed calcium is now determined by the original process. Soil type and the presence of zeolitic material affect the value of the process, and soils which appear neutral by a number of chemical processes still show a lime requirement in field trials. A. G. Pollard.

Determination of the lime requirement of some Brunswick soils. A. Gehring, A. Peggau, and O. Wehrmann (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, A, 153—169).—The lime requirement of a number of soils was calculated from (1) a determination of replaceable calcium by extraction with sodium chloride, and (2) the maximum calcium adsorption obtained by a similar process after saturating the soil with lime water. On this basis soils showing 70% saturation with lime gave no increased crop yields after liming, provided little or no hydrolytic acidity was observed in the original soil. The degree of saturation with lime showed a fairly close inverse relationship to crop increases following requisite liming. The discrepancy frequently observed between the results of field trials and the determination of lime requirements by the usual chemical processes is discussed.
A. G. Pollard.

Effect of regulated treatment with hydrochloric acid on the lime requirement of a mineral subsoil. G. Milne (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, A, 126—133).—Extraction of soil by hydrochloric acid solutions of increasing strength (0·02—5·5*N*) showed that following the weaker acid extractions the lime requirement of the soil residue increased to a maximum and then steadily decreased as more concentrated acid was used. The initial increase in lime requirement is attributed to the replacement of exchangeable basic ions by hydrogen ions. The subsequent decrease in lime requirement represents the steady decomposition of colloidal matter. A. G. Pollard.

Reaction between soils and hydroxide solutions. S. J. Saint (Trans. 2nd. Comm. Int. Soc. Soil Sci., 1926, A, 134—148).—The absorption of bases by soil is measured by treatment with alkaline-earth hydroxides of varying concentration. Means for determining the " base requirement " of soils from these measurements are described. When the hydroxides of sodium and potassium are used the results differ from those obtained with alkaline-earth hydroxides by an amount which is constant for any particular soil. The method may be used

to measure replaceable hydrogen ions in the soil. Increased absorption of bases after extracting the soil with very dilute acid is equivalent to the replaceable bases removed by this means. The " base requirement " is unaffected by the ratio of soil to solution used, but is slightly increased by the time of contact and the fineness of grinding of the soil. Evidence is advanced to show that the soil colloids are not flocculated directly by the hydroxides of sodium and potassium, but that flocculation results from the activity of the exchangeable calcium ions so displaced. A. G. Pollard.

Relation between soil reaction and plant growth. L. von Kreybig (Trans. 2nd. Comm. Int. Soc. Soil Sci., 1926, A, 149—152).—The range of p_H value of a soil for optimum plant growth is not an absolute figure. In soils exhibiting hydrolytic or exchange acidity (Kappen) or having a strong reducing power for permanganate the optimum ranges may vary considerably. Lucerne is a particularly sensitive crop in this respect. Successful crops are only produced on soils having a high buffer capacity. A. G. Pollard.

Investigations of K. K. Gedroiz on base exchange and absorption—a *résumé*. H. J. Page (Trans. 2nd Comm. Int. Soc. Soil Sci., 1926, A, 208—231).—The theories of the mechanism of base exchange, clay flocculation, energy of base absorption, etc. are based on the conception that a replaceable base in the soil can be completely replaced by another base, but the process is only complete after repeated treatment with a salt solution. Details of the process for carrying out this replacement in the laboratory are given and adaptations for alkali soils are indicated. A. G. Pollard.

Losses of phosphoric acid by leaching from upland soils in North Wales. G. W. Robinson and J. O. Jones (Agr. Progress, 1926, 3, 39—42).—Phosphate added as basic slag or Gafsa phosphate to soils devoid of calcium carbonate gradually disappears from the upper layers, and is precipitated in the lower layers, probably in combination with hydrated sesquioxides. Extraction with $0\cdot2N$-nitric acid appeared to give a degree of discrimination as to the combination of the phosphoric oxide which was not attained by extraction with 1% citric acid solution. Chemical Abstracts.

Carbon dioxide production in soil and solution. D. V. Bal (Ann. Appl. Biol., 1926, 13, 231—243).—Dextrose, lævulose, sucrose, lactose, and maltose are decomposed by *Bacillus prodigiosus* in decreasing order of readiness; ethyl alcohol, acetone, and organic acids are produced in addition to carbon dioxide. The maximum production of carbon dioxide is attained in 3—4 days. The lowering of the carbon dioxide production in soil is not due to exhaustion of the total available carbon, the formation of a film on the particles of organic matter, or the exhaustion of available mineral constituents. Addition of organic matter to soil, after carbon dioxide production has declined, restores the process to its initial level. Chemical Abstracts.

Nature of the colloidal soil material. P. L. Gile (Third Colloid Symposium Monograph, 1925, 216—227).—Colloidal soil material consists chiefly of silica, alumina, iron, organic matter, water not removed at 110°, and smaller quantities of magnesium, potassium, calcium, titanium, sodium, phosphorus, and manganese. The colloid is not wholly amorphous. A dispersed particle of soil colloidal material acts like a loose mosaic of mixed ingredients with an internal pore space. Replaceable bases are mainly held at the surfaces presented by silica and organic matter. Chemical Abstracts.

Influence of soil, season, and manuring on the quality and growth of barley. IV. E. J. Russell (J. Inst. Brew., 1927, 33, 104—110. Cf. B., 1925, 1002).—The influence of nitrogenous manures has been practically the same as in previous years. The use of ammonium sulphate has not adversely affected the quality and has increased the yield. On an average, the addition of 1 cwt. of ammonium sulphate increased the yield of total grain by 7·4 bushels per acre. The nitrogenous manures, however applied, almost always raised the percentage of nitrogen in the grain. Ammonium chloride can be safely used as a substitute for ammonium sulphate. In addition to increasing the yield and relatively lowering the nitrogen content of the grain, it increases the number of grains per acre reaching the standard of head corn by reducing the number of infertile florets or of immature corns. The effect of phosphate has been to give a more marked increase than usual, whilst potassium sulphate was without effect. C. Ranken.

Unsulphonated residue in spray oils. Graham.—See II.

Decomposition of cellulose in soil. Winogradsky.—See V.

Nitrogen in cocoa by-products. Walton and Gardiner.—See XIX.

Patents.

Treatment of calcium cyanamide. G. Barsky, Assr. to American Cyanamid Co. (U.S.P. 1,618,047, 15.2.27. Appl., 29.12.23).—Finely-divided calcium cyanamide (100 pts.) is mixed with water (200 pts.), and added to sulphuric acid (d 1·71, 900 pts.). Decomposition of the cyanamide to carbamide and ammonium sulphate proceeds smoothly without formation of by-products, *e.g.*, dicyanodiamide. Phosphate rock (1000 pts.) can then be added if desired. T. S. Wheeler.

Removing dust from calcium cyanamide and deodorising the same. J. Breslauer, Assr. to Comp. de l'Azote et des Fertilisants Soc. Anon. (U.S.P. 1,619,185, 1.3.27. Appl., 8.1.26. Conv., 10.1.25).—The cyanamide is simultaneously treated with a current of carbon dioxide and superheated steam for a period insufficient to cause any marked evolution of ammonia. H. Royal-Dawson.

Compost manures. F. Mühlbachl (Austr. P. 103,913, 28.12.25).—Bones and combustible material are separated from domestic refuse. The combustible material is burnt and the ashes are mixed with the bones. The mixture is moistened with dilute sulphuric acid, and turned over from time to time with fresh additions of acid. Other refuse, including excreta, mixed with calcium carbonate is added, and the mixture is allowed to ferment. G. W. Robinson.

Fertilisers. ELEKTRIZITÄTSWERK LONZA (GAMPEL U. BASEL), and E. LÜSCHER (Swiss P. 116,162—3, 3.3.25).—A solution containing phosphoric acid and ammonia in the proportions requisite for the formation of diammonium phosphate is atomised in a stream of air containing ammonia and water vapour. The phosphoric acid solution can also be absorbed in peat, and two layers can be treated with a current of air containing ammonia, until the lower layer contains ammonia in excess of that required for the formation of diammonium phosphate. It is then treated with a current of air almost free from ammonia. The phosphoric acid solutions may be obtained by extracting superphosphate with water or with carbamide solutions. Dicyanodiamide present in crude carbamide solutions is changed by the acid of the superphosphate extracts into guanylcarbamide. G. W. ROBINSON.

Insecticide. H. HOWARD, Assr. to GRASSELLI CHEMICAL Co. (U.S.P. 1,620,208, 8.3.27. Appl., 15.9.24).—Barium silicofluoride is claimed as an insecticide. G. W. ROBINSON.

Copper-lime dust. G. E. SANDERS, Assr. to RICHES, PIVER & Co. (U.S.P. 1,620,490, 8.3.27. Appl., 9.8.24. Cf. U.S.P. 1,541,753; B., 1925, 689).—A fungicide is prepared by adding copper sulphate crystals to quicklime, whereby the copper salt becomes dehydrated. G. W. ROBINSON.

Plant sprays. I. G. FARBENIND. A.-G., Assees. of A. STEINDORFF and H. MEYER (G.P. 435,389, 19.12.22. Addn. to G.P. 433,653; B., 1927, 233).—Plant sprays are prepared consisting of halogenated naphthenic acids or their salts mixed, if desired, with hydrocarbons or their halogen substitution products. The halogenated naphthenic acids have a weaker odour and are less harmful to plants than the unsubstituted acids mentioned in the earlier patent, whilst their insecticidal action is increased. They have an emulsifying effect on the hydrocarbons or their derivatives, which also increase the insecticidal effect. G. W. ROBINSON.

Dusting powder adherent to plants. R. LIESKE, A. THAUSS, and W. BONRATH, Assrs. to WINTHROP CHEMICAL Co., INC. (U.S.P. 1,616,323, 1.2.27. Appl., 25.9.25. Conv., 3.4.25).—The addition of a small quantity (0·3%) of a condensation product of cellulose and a halogenated aliphatic acid, such as is described in G.P. 332,203 (B., 1921, 344 A), to an insecticidal powder for plants greatly improves its adherence. T. S. WHEELER.

XVII.—SUGARS; STARCHES; GUMS.

Starch nitrate. H. OKADA (Cellulose Ind., Tokyo, 1927, **3**, 3—14).—With suitable cooling and stirring there is no difficulty in introducing the starch into the mixed acids and obtaining nitrates in theoretical yields. Stabilisation is readily effected by washing the nitrate with hot, strong, aqueous alcohol, to which may be added a small quantity of acetone. The starch is regenerated quantitatively by denitration with alcoholic ammonium hydrosulphide of low concentration and thoroughly cooled. The sulphur is removed from the product by careful treatment with hydrogen peroxide, hot alcohol, and hot acetone. The regenerated starch has a low copper value of 1—3; it gives a blue reaction with iodine, and a clear solution in cold water. Thus it represents the ideal soluble starch, only physically modified, without chemical hydrolysis or oxidation. The washing and stabilisation of the starch nitrate are facilitated by swelling the granules. The swelling action of alcohol, alcohol–acetone, and water–acetone was studied; water–acetone is the most suitable agent. In spite of the higher density of starch as compared with cellulose, starch nitrate shows no higher content of sulphuric esters than does nitrocellulose. J. F. BRIGGS.

Rapid method for determination of starch. O. S. RASK (J. Assoc. Off. Agric. Chem., 1927, **10**, 108—120).—Treatment with cold hydrochloric acid renders starch completely water-soluble and subsequently coagulable by alcohol. There is an optimum time, temperature, and acid concentration relationship to secure complete conversion without hydrolysis. The action of acid is made the basis of a method for the determination of starch. 1—4 g. of material are ground, transferred to a filter paper, and successively extracted with ether, 10% alcohol, and water in the order named. The paper and residue are then stirred with 20—25 c.c. of dilute hydrochloric acid. The suspension is diluted to 100 c.c. and filtered through dry asbestos. (The residue should show no starch reaction.) 50 c.c. of the filtrate are pipetted into 110—115 c.c. of 95% alcohol, stirred for 1 min., and the precipitate allowed to settle. The flocculate is filtered, washed with 96% alcohol, and dried to constant weight. (The filtrate should show no reaction of carbohydrates by the Molisch test.) Application of the method to a variety of starches and cereal products revealed that it determines starch, and starch only, with a high degree of accuracy. The results are considerably higher than those given by the diastase method. H. DOWDEN.

Relative sweetening power of sucrose and lævulose. O. SPENGLER and A. TRAEGEL (Z. Ver. deut. Zuckerind., 1927, 1—12).—Tasting tests in which 8 or 9 persons participated gave the following values for the two sugars: 100 g. of lævulose = 108 g. of sucrose, the values being approximately the same when the solutions were made with distilled water, tap water, or tap water with an addition of 0·01% of sodium chloride. J. P. OGILVIE.

Clarification for polarisation. R. T. BALCH (Sugar, 1926, **28**, 551).—Hydrated aluminium silicate is ground to an impalpable powder in a ball mill, and mixed with distilled water to form a 5% suspension, the water being added in small portions at first, and thoroughly mixed after each addition until the tendency to lump is overcome, after which the remainder of the water is added in a single portion. For most purposes 5 c.c. of this suspension are sufficient for the clarification of 100 c.c. of solution for polarisation. It fulfils requirements much more effectively than the alumina cream usually added, not only assisting the rate of filtration, but also effecting some decolorisation. J. P. OGILVIE.

Bagasse as fuel. R. F. HUTCHESON (Int. Sugar J., 1926, **28**, 652—658).—Assuming the following principal data: cane crushed per hr., 50 tons; fibre in cane,

16%; bagasse in cane, 34%; fibre in bagasse, 47%; sugar in bagasse, 4%; water in bagasse, 48%, the following values are calculated: actual heat absorbed by water in the boilers, 2702 B.Th.U.; fuel efficiency, 75%; volume of flue gases, 115 cub. ft., and boiler h.p., 2266. J. P. OGILVIE.

Determination of cuprous oxide produced in sugar analysis. C. S. BISSON and J. G. SEWELL (J. Assoc. Off. Agric. Chem., 1927, **10**, 120—124).—Cuprous oxide formed by the reduction of Fehling's solution, after filtration and washing, is completely oxidised by a measured volume of standard permanganate solution. The precipitate is dissolved in sulphuric acid (*d* 1·495), and a measured volume of standard ferrous sulphate solution added, in excess of that required to destroy the colour of the permanganate. After dilution to standard volume, the excess ferrous sulphate is titrated with permanganate to the usual end-point. The method is an improvement on that of Caven and Hill (cf. B., 1897, 981; 1898, 124). H. J. DOWDEN.

Determination of sulphur dioxide in sugar factory products. J. P. OGILVIE (Int. Sugar J., 1926, **28**, 644—647).—Very sensitive results are yielded by the sulphide stain method, using modifications communicated by J. S. MANN, in which a flask is provided with a thistle funnel and a tap funnel, and a leading tube to which is attached a glass tube holding a small cotton plug, and a funnel, this latter having a clamp for holding filter-paper impregnated with lead acetate. About 50 g. of purest granulated zinc (previously washed with dilute hydrochloric acid to remove traces of sulphur always present) are placed in the flask, just covered with water, and 50 c.c. of pure dilute hydrochloric acid (1 : 2 of water) are run slowly from the tap funnel through the thistle funnel over the zinc, the gases evolved being passed through the lead-impregnated paper for 30 min., at the end of which time no discoloration of the paper should have resulted. A series of standard stains are prepared by the introduction into the flask of known amount of sodium sulphite dissolved in pure sugar liquor, so as to represent quantities of 0·0025, 0·0020, 0·0015, and 0·0010% SO_2 in samples of a sugar product when 5 g. are taken for the determination. J. P. OGILVIE.

Apparatus for the electrometric determination of ash in sugar factory products. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1927, **51**, 205—211).—Instead of using a Wheatstone bridge with telephonic indication, the author utilises alternating current from the lighting main divided into four resistances, two of which are lamp bulbs, the others being the liquid under examination and a constant resistance. By altering the distance between the electrodes in the cell containing the assay liquid, the resistance is altered and balanced with the constant resistance, the two lamps being equally illuminated at the point of equilibrium. An apparatus constructed on this principle is said to have yielded sufficiently precise results in the electrometric determination of mineral matter in sugar products, the difference between them and the values given by the gravimetric method in the case of raw sugars being less than 0·02% in 68% of the samples examined, and exceeding 0·04% only in four cases. J. P. OGILVIE.

Fermentable sugars of malt. WRIGHT.—See XVIII.

PATENTS.

Absorbent decolorising medium. C. B. DAVIS (U.S.P. 1,618,149, 15.2.27. Appl., 14.12.23).—A porous carbon base such as carbonised bones, carbonised gel of raw sugar gums, or the exhausted carbon used in decolorising sugar liquors, after cleaning with dilute sodium hydroxide solution and hydrochloric acid, is heated at 250—1000° with the pyro-bituminous gas from the "cracking" of heavy oil distillates, in the presence of a limited amount of oxygen. The hard, finely-divided carbon structure formed is strongly absorbent. F. R. ENNOS.

Purifying carbohydrate solutions etc. C. B. DAVIS (U.S.P. 1,618,148, 15.2.27. Appl., 21.2.23).—By the addition of a small, predetermined quantity of colloidal tannate of iron to the carbohydrate solution at 70°, impurities consisting of electro-positive colloids are neutralised and precipitated. After removal of the precipitate by a filter press, the liquid is decolorised by passing through a char filter. F. R. ENNOS.

Manufacture of an adhesive. L. FACKLER, Assr. to STEIN-DAVIES Co. (U.S.P. 1,618,150, 15.2.27. Appl., 15.12.21).—A colourless liquid adhesive is made by mixing dry dextrin with dry sodium perborate, and subsequently adding water. The oxygen evolved bleaches the dextrin, and the residual boron compound accelerates its dissolution. F. R. ENNOS.

XVIII.—FERMENTATION INDUSTRIES.

Influence of the hydrogen-ion concentration on the velocity of the fermentation and yield of alcohol. J. DEHNICKE and W. KILP (Z. Spiritusind., 1927, **50**, 69—70).—Worts of varying hydrogen-ion concentration prepared from maize and from potatoes were fermented after adjustment to equal extract contents. The highest yields of alcohol were obtained with an initial p_H 4·6—5·3 for the maize worts and 5·0—5·5 for the potato worts, and these yields were attained at a stage of the fermentation on an average, 20 hrs. earlier than with worts having p_H values outside those limits. Worts prepared similarly from molasses gave irregular results, which were probably due to the greater salt content of the molasses. C. RANKEN.

Influence of soil, season, and manuring on the quality and growth of barley of the 1925 crop as indicated by the malts made therefrom. IV. H. M. LANCASTER (J. Inst. Brew., 1927, **33**, 111—119; cf. B., 1926, 103).—The malting loss, malt extract, and valuation of the barley were the points taken for comparison of the barleys. The malting loss was calculated on raw barley so that any loss of dry matter incurred during the sweating process was included. The malts were all made from barley of the same pure variety of seed, and whilst the similarity of the malts from the same farm under different manurial conditions was remarkable, the difference due to soil and climate was very great. In general malting value, Spratt–Archer and Archer–Goldthorpe were slightly superior to Beaven's Archer and Webb's Sunrise, but all these varieties produced good malting barleys. The results

of the analyses of the malts described are recorded in an appendix by H. L. Hind. C. Ranken.

Strength of yeast cells. S. B. Schryver, E. T. Thomas and S. G. Paine (J. Inst. Brew., 1927, **33**, 120—137).—The softening of yeast (autolysis) does not depend upon the state of the cell walls, but is the result of chemical changes, which seem to be bound up with the nitrogen content and its distribution in the cell. During the softening of yeast there is an increase in the amount of material which can be extracted with water, and the amount of protein diminishes with an increase in the non-protein content. Following a latent period there is a rapid degradation of the protein which is not due to bacteria, which only find a suitable pabulum after autolysis has started. Autolysis, which takes place only in acid media, is inhibited by higher concentrations of the acid. No autolysis occurs if the yeast is thoroughly washed in water and allowed to remain in a wet condition. The water removes some product which is essential to autolysis and concentrated washings from yeast accelerate autolysis. The washings apparently contain one or more proteolytic enzymes. Pressure on the yeast also favours autolysis. C. Ranken.

Fermentable sugars of malt. I. G. S. Wright (J. Inst. Brew., 1927, **33**, 151—170).—The soluble constituents of malt include, in addition to sucrose, dextrose, and lævulose, a series of fermentable polysaccharides which are products of the malting process. Among these polysaccharides a carbohydrate which may have the structure of a dextrose–lævulose–galactose trisaccharide is indicated, also a gluco-glucoside and other carbohydrates in which dextrose, lævulose, and galactose are constituted. No indications of maltose were afforded in any of the malts examined either by the osazones or considerations of the reducing and rotatory powers. Notes on the determination of the cold-water extracts of malt are appended. C. Ranken.

Manuring experiments on hops. Burgess.—See XVI.

Carbon dioxide production. D. V. Bal.—See XVI.

XIX.—FOODS.

Reductase test. E. R. Hiscox (Agr. Progress, 1926, **3**, 102—103).—The reductase test gives erratic results, and is inferior to the plate count method for grading milk according to the English system.

Chemical Abstracts.

Technique of the Gerber test. J. Golding and A. Wagstaff (Agr. Progress, 1926, **3**, 103—104).—Sulphuric acid (*d* 1·820—1·825, 10 c.c.) is automatically measured into a butyrometer bottle, and amyl alcohol (*d* 15·5 0·8145—0·816, 1 c.c.) is run on to the surface; milk (11 c.c.) is added (not using a pipette with a wide delivery tube), the bottle is closed with a grey rubber stopper, wrapped in cloth, shaken by hand, and centrifuged for 3 min. Before reading off the fat, the bottle is placed in water at 65·5°. Chemical Abstracts.

Technique in testing for vitamin-*B*. A. L. Bacharach and G. A. Hartwell (Analyst, 1927, **52**, 145—149).—The weight curves of rats on vitamin-*B*-free diet did not vary for two separate inbred stocks, and similar curves were obtained by two workers in different laboratories with their own and each other's animals. In order to test for the absence of vitamin-*B*, the quickest results were obtained by using "raw" rather than "cooked" food; the latter method is, however, a more sensitive way of showing its presence. D. G. Hewer.

Recent advances in the bacteriological examination of food and water. W. G. Savage (Analyst, 1927, **52**, 117—127).—The correlation of hydrogen-ion concentration in water, as determined by the use of methyl red, with the Voges and Proskauer and the Koser reactions, has been studied, and a division of the lactose-fermenting bacilli into high and low types was found to correspond with primary and, possibly, fundamental differences. Detection of tubercle bacilli and problems in connexion with streptococci are discussed, together with laboratory methods for judging the extent of clean milk production. Details in connexion with the investigation of outbreaks of food poisoning with no ascertainable cause are considered, and a systematic method of examining canned foods is outlined, the range of culture media and incubation temperatures sufficient to include all types of spoilage organisms being indicated.

D. G. Hewer.

Analysis of flours. G. Smirich (Annali Chim. Appl., 1927, **17**, 81—82).—The separation of fragments of husk etc. from flour, bread crumb, and the like, for microscopical identification may be effected by heating 5—10 g. of the material for 5—10 min. with 50—100 c.c. of 85% glycerol containing 20 g. of concentrated sulphuric acid per litre. The hot liquid is centrifuged, and the residue of bran etc. examined. T. H. Pope.

Relation of the magnesium in the ash and the lipoid–protein ratio to the quality of wheats. B. Sullivan and C. Near (J. Amer. Chem. Soc., 1927, **49**, 467—472).—Wheat of 20 varieties (of strain or place of growth) was examined with regard to protein content, gluten content and quality, proportion and constituents of ash, and lipoid content. The magnesium content of the ash has a direct relationship to the strength of the wheat as determined by protein content and quality of the gluten. The latter was not related to the calcium, potassium, and phosphorus contents of the ash. The lipoid contents were all between 2·53% and 3·84%. Softer wheats show a higher ratio of lipoid to gluten or lipoid to protein. F. G. Willson.

Significance of solubility and "activity" of nitrogen in cocoa by-products. G. P. Walton and R. F. Gardiner (J. Assoc. Off. Agric. Chem., 1927, **10**, 135—142).—The water-soluble nitrogen content, which has been taken as a criterion of fertiliser value, has been determined for the cocoa by-products, press-cake, solvent-extracted cocoa, and cacao shell meal. The total content of nitrogen in dry fat-free press-cakes and solvent-extracted cocoas averages just under 5%, of which one third is water-soluble. 50—58% of the water-soluble nitrogen is represented by theobromine and caffeine, the fertiliser value of which compounds is questionable. The "activity" or ease with which water-insoluble nitrogen is rendered water-soluble, as determined for press-cake by the neutral permanganate method, was found to be above the acceptance value of

80%, but by the alkaline permanganate method to be less than one half of the "passing" limit of 50%. The validity of the "activity" determination is dependent upon rigid adherence to the standard procedure, for drying the water-insoluble portion at 70° prior to the neutral permanganate treatment decreased the "activity" from 89·7% to 76·7%. H. J. DOWDEN.

Determination of caffeine in de-caffeinised coffee. G. BONIFAZI (Ann. Chim. Analyt., 1927, [ii], 9, 33—38).—25 g. of coffee and 5 c.c. of 5% ammonia solution are extracted for 3—4 hrs. with chloroform or ether, the solvent is distilled off, the residue extracted with cold water, and the solution boiled with 1 g. of magnesia. After filtering and washing the residue with boiling water, the filtrate is evaporated to dryness, taken up in boiling water, and 5 c.c. of 1% potassium permanganate solution are added. The solution is decolorised with 3% hydrogen peroxide containing 1 c.c. of glacial acetic acid per 100 c.c. The residue is extracted with boiling chloroform. When the chloroform is nearly evaporated the solution is transferred to a subliming capsule, the residue of solvent evaporated, and the sublimed product weighed. Purification of the caffeine may be further effected by a second treatment with permanganate, or by successive sublimations. D. G. HEWER.

Analysis of egg yolk preserved with glycerin. T. COCKBURN and M. McF. LOVE (Analyst, 1927, 52, 143—144).—Moisture is determined by drying *in vacuo* over sulphuric acid for 48 hrs.; fat by the Gottlieb method, since chloroform cold or hot dissolves appreciable amounts of glycerin; and glycerin by making 10 g. of the sample into a thin paste with water at 45—50°, and adding dialysed iron (B.D.H.) drop by drop until the proteins are precipitated. The mixture is filtered, the filtrate and washings are made up to 250 c.c., and the glycerol is determined in 100 c.c. by the acetin method. The approximate proportion of glycerin present may be arrived at by shaking some of the above filtrate twice with ether to remove fat, adding the water washings of the ethereal solution to the original aqueous layer, evaporating to dryness, and weighing. D. G. HEWER.

Variations in the composition of Colorado potatoes. N. E. GOLDTHWAITE (Colorado Agric. Exp. Sta. Bull., 1925, [296], 3—77).—No two individual potatoes have the same composition. The percentages of dry matter, starch, and total carbohydrates vary inversely with the percentage of water. There is little relationship between the nitrogenous matter and ash. For irrigated potatoes, the percentage of dry matter is approximately 6·71% of the total greater than that of the starch, the relation being 1·42 : 1, and that of the total carbohydrates to dry matter 1 : 1·15. The ratios starch : water and total carbohydrates : water are very approximately 1 : 1·5 and 1 : 3·897. On the dry basis, the ratio starch : dry matter is 1 : 1·25.
CHEMICAL ABSTRACTS.

Determination of starch.—RASK.—See XVI.

PATENTS.

Treating milk products. U. G. TODD, Assr. to PFAUDLER CO. (U.S.P. 1,618,789, 22.2.27. Appl., 20.5.24).—Milk, the acidity of which has been adjusted so as to prevent coagulation in the subsequent process, is heated at 60—65°, and subjected, while in a finely-divided state obtained by spraying, to the purifying action of a current of steam or steam and air in a partial vacuum, whereby undesirable flavours and odours are removed.
F. R. ENNOS.

Production of dry milk. W. B. MCLAUGHLIN (U.S.P. 1,616,631, 8.2.27. Appl., 31.3.23).—Milk, with the addition of 2% of sugar, is concentrated under reduced pressure to d 1·1, heated to the b.p. by passing steam into it, and maintained under the influence of its own contained heat for 1 hr. in a heat-insulated tank. After spray-drying the treated milk, a product is obtained of altered flavour, improved solubility, and better keeping qualities. F. R. ENNOS.

Producing commercial baking dough. D. S. DONOVAN and J. R. BROWN (U.S.P. 1,617,514, 15.2.27. Appl., 6.7.26).—Sponge dough ingredients, including the yeast, are mixed and allowed to remain for 2 hrs. at 24—27°, so that fermentation is substantially complete. Soda and baking powder are then added to the mixture, which is cooled, preferably in small lots, to 0—2° before the aerating agent can react, and maintained in this condition until ready for baking. F. R. ENNOS.

Manufacture of biscuits, cakes, and similar food products. O. A. ELIAS (E.P. 266,101, 12.1.26).—The baking process is carried out in the presence of artificially-produced ultra-violet rays. The temperature of the oven, which is heated by both light and radiant heat, is maintained at about 90°, although it may be raised for a short period to 200°. The product has an increased food value. F. R. ENNOS.

Preserving food products. J. J. BARRY, Assr. to GENERAL SEAFOODS CORP. (U.S.P. 1,617,006, 8.2.27. Appl., 1.10.24).—Maize or other food product is stirred and heated at 100° while the air is withdrawn and replaced by carbon dioxide under pressure. After reduction of the pressure, the cooked food product is transferred to containers, which are sealed and heated at the sterilising temperature (112°) for 30 min. F. R. ENNOS.

Treatment of vegetable produce. S. G. M. NESBITT, J. B. BUTLER, and J. J. DRUMM (E.P. 266,069, 30.11.25).—The vegetables are boiled with water and steeped in a non-toxic, alkaline buffer solution (4—8% solution of crystalline disodium phosphate). They are then packed in sealed containers with the usual sugar–salt solution, together with enough buffer solution so that after sterilisation by heat the product has p_H 7·3—8·3, when the original green colour is preserved. F. R. ENNOS.

Method and apparatus for sterilising and preserving food. R. KRISTEN (E.P. 265,859, 26.7.26).—The food contained in preserve glasses is inserted in a vessel with an air-tight fitting lid, and a small quantity of alcohol in the vessel is exploded by means of an ignition device operated from the outside. The oxygen in the vessel and in the preserve glasses, the lids of which are slightly lifted by the explosion, is thus rapidly burnt and the harmful germs are killed, the combustion gases being expelled into a collecting rim surrounding the air-tight lid, which is lifted against spring pressure. Admission of air into the vessel instantaneously closes the lids of the glasses, which are then removed.
F. R. ENNOS.

Preparation of a food-colouring composition. W. D. Bost, Assr. to Orange Crush Co. (U.S.P. 1,616,739, 8.2.27. Appl., 18.7.21).—A colouring compound for potable liquids is prepared by heating carmine with alkaline aqueous glycerin. After intensifying the colour by boiling with concentrated ammonia solution, the product is rendered slightly acid by boiling with phosphoric acid, and, before use, is diluted with sugar solution (*d* 1·21 or over). F. R. Ennos.

Improving the qualities of coffee. O. Gewalt (E.P. 265,865, 16.8.26).—The coffee, preferably when green, is subjected to a temperature of about −8° for 20 hrs. or more, when any unpleasant odour and taste are removed. F. R. Ennos.

Pulverising and separating cereals (E.P. 265,841). —See I.

Vitaminised oils (E.P. 266,139).—See XII.

Sterilising putrescible liquids (E.P. 254,725).—See XXIII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Titration of tartaric acid after drying at 100°. A. Engler (Chem.-Ztg., 1927, **51**, 158—159).—Degener (B., 1898, 700) has stated that tartaric acid after drying at 100—105° suffers a diminution of titration acidity, presumably owing to the formation of anhydride. The author repeated this experiment using chemically pure tartaric acid dried at 105° for 17 hrs. It was then titrated with 0·533*N*-caustic potash with phenolphthalein or litmus paper. Identical results were obtained at 0° and at room temperature. By heating at 95° the neutral solution was rendered very faintly acid. It is concluded that Degener's results were erroneous. C. Irwin.

[Determination of] ethyl phthalate. IV. J. A. Handy and L. F. Hoyt (J. Amer. Pharm. Assoc., 1926, **15**, 454—461).—The sample (0·1 c.c.) is heated on a steam bath with ethyl-alcoholic potassium hydroxide free from aldehydes (1 c.c.) until the alcohol is completely removed, the resorcinol–sulphuric acid reagent (0·5 c.c.) added, and the mixture heated at 150° for 3 min., then diluted with 40 c.c. of water and rendered alkaline with 10 c.c. of 10% sodium hydroxide solution to produce the fluorescence. The addition of 1 c.c. of alcoholic potassium hydroxide to 1 c.c. of the sample containing 0·5% or more of ethyl phthalate yields crystalline potassium phthalate. By using 10 c.c. of the sample and 1 c.c. of alcoholic potassium hydroxide, 0·05% of ethyl phthalate may be detected. Chemical Abstracts.

Errors in analysis of alkaloids caused by presence of fatty acid or soap. H. R. Watkins and S. Palkin (J. Assoc. Off. Agric. Chem., 1927, **10**, 130—135. Cf. B., 1926, 848).—Determination of alkaloids by chloroform extraction is often vitiated by the presence of fatty acid or soap, especially when calcium or magnesium compounds are present. Potassium soaps lead to the formation of difficult emulsions, rendering the extraction incomplete. Calcium and magnesium soaps, being slightly soluble, are extracted simultaneously with the alkaloid and give rise to a high assay. Ammonium soaps are hydrolysed and the free acid is extracted, to be deposited on the walls of the vessel during the final titration, but without influencing the determination. Sodium soaps, however, in small quantities do not give this deposit. When soap settles out during the final titration, a few c.c. of 0·1*N*-acid should be added, and the fat removed by extracting twice with chloroform, the aqueous alkaloidal solution being then made ammoniacal and re-extracted. H. J. Dowden.

Analysis of sodium salicylate and sodium benzoate. D. Henville (Analyst, 1927, **52**, 149—150).—The weighed sodium salicylate is transferred to a separator, neutral ether and methyl orange are added, and 0·5*N*-hydrochloric acid is run in until the indicator is a distinct red. The aqueous layer and ether washings are run off and neutral ether is added, when, on shaking, the indicator reverts to yellow. Titration is continued to the end-point, which is quite sharp. In the presence of sodium or potassium carbonates or bicarbonates, the total combined alkali is obtained as above, the carbonates are removed, the solution is filtered, washed, and titrated as before, or the boiling solution is titrated with standard acid, the figure calculated to carbonate or bicarbonate, and the difference from the original ether titration figure calculated to sodium salicylate. D. G. Hewer.

Microchemical examination of santonin and herapathite [quinine periodide]. C. van Zijp (Pharm. Weekblad, 1927, **64**, 278—284).—If a sublimate of pure santonin be covered with water, it slowly dissolves. On evaporation of the water, the santonin separates in oily drops, but if the glass be scratched before evaporation, or seeded, the crystalline form separates again readily. A solution of the sodium compound gives crystalline deposits readily with calcium, strontium, cadmium, cobalt, or lead acetates. Conditions for microchemical examination using these reactions are described. Herapath's reaction is suitable for microchemical identification of quinine; suitable conditions by which the test may be carried out, even in the presence of twice the quantity of cinchonidine, are described. S. I. Levy.

Humic acids. I. Methylation and treatment with ammonia of some humic acid samples. W. Fuchs and H. Leopold (Brennstoff-Chem., 1927, **8**, 73—77).—Three samples of humic acid, viz., Merck's humic acid, and humic acid prepared from starch and from quinol, were subjected to repeated methylation (with methyl sulphate and alkali) and treatment with ammonia. The products from the three samples gave, respectively, the following limiting values: methoxyl content, 6·9%, 7·6%, 2·5%; total nitrogen, 7·50, 5·95, 11·46; nitrogen removable with alkali, 4·20, 3·15, 4·53; fixed nitrogen, 3·30, 2·80, 6·93. Reckoning with a mol. wt. of 1400, the molecule of humic acid contains only one phenolic hydroxyl group to not less than twenty carbon atoms. Exhaustive methylation considerably reduces the reactivity of the samples towards ammonia, and *vice versa*. This observation and the hypothesis that coal is formed from lignin *via* humic acids and humins, are briefly discussed. W. T. K. Braunholtz.

[Composition of plants.] E. V. LYNN and P. Y. CHENG (J. Amer. Pharm. Assoc., 1926, **15**, 105—108).—An examination of *Lysichiton camtshatcene* (skunk cabbage), *Asarum caudatum* (wild ginger), *Gaultheria shallon* (salal), and *Micromeria douglassi* (tea vine). Wild ginger contains a small amount of volatile oil, f.p. 4—5°, n_D^{20} 1·5195. CHEMICAL ABSTRACTS.

Essential oils of *Mentha aquatica* and *M. sylvestris* from Sicily. G. ROMEO and U. GIUFFRÈ (Annali Chim. Appl., 1927, **17**, 83—87).—Oil of *Mentha aquatica* from Messina has d^{15} 0·9671, α_D^{20} 39° 55′, n^{20} 1·4883, solubility in 75% alcohol, 1 : 1·5, acid value 1·45, saponification value 64·85, esters (as menthyl acetate) 22·41%, combined alcohols (as menthol) 17·66%, free alcohols 28·53%, ketones (as menthone) 0·77% (cf. Kremers, B., 1922, 647A). Oil of *Mentha sylvestris* from Messina has d^{15} 0·9687, α_D^{20} + 20° 15′, n^{20} 1·4841, solubility in 75% alcohol 1 : 1, acid value 1·40, saponification value 82·6, esters (as menthyl acetate) 28·71, combined alcohols (as menthol) 22·62%, free alcohols 30·84%, ketones and aldehydes, trace (cf. Schimmel's Report; B., 1910, 716). T. H. POPE.

Essential oils of *Calamintha nepeta* and *Mentha pulegium*. G. ROMEO and U. GIUFFRÈ (Annali Chim. Appl., 1927, **17**, 87—88. Cf. B., 1926, 107).—Oil of *Calamintha nepeta*, var. *Canescens* (*Mentha pulegium*, var. *tomentosa*), obtained in 0·7 (0·9)% yield from the dry plant, has d^{15} 0·9218 (0·9262), α_D^{20} + 20° 10′ (+38° 40′), α_D^{20} 1·4778 (1·4733), solubility in 75% alcohol 1 : 1·5 (1 : 1·25), acid value 1·12 (1·55), saponification value 7·03 (6·8), esters (as menthyl acetate) 2·09 (1·85)%, combined alcohols (as menthol) 1·64 (1·46)%, free alcohols 16·67 (10·23)%, ketones (as pulegone) 56·10 (70·54)%. T. H. POPE.

Pyrogenic dehydration of fusel oil. M. GIUA and L. THUMINGER (Atti R. Accad. Sci. Torino, 1926, **61**, 149—158). See B., 1926, 687.

PATENTS.

Production of methane. CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 244,076, 19.11.25. Conv., 3.12.24).—Methane is produced from carbon monoxide and hydrogen or gas mixtures containing hydrogen, by means of nickel catalysts, the rate of reaction being increased and the use of smaller contact furnaces or of a smaller amount of contact substance being made possible by working at atmospheric pressure and at temperatures above 500°. C. O. HARVEY.

Preparation of concentrated acetic acid from dilute aqueous or crude dilute pyroligneous acid. H. SUIDA (E.P. 255,043 and 255,047, [A] 21.6.26, [B] 24.6.26. Conv. [A, B], 13.7.25).—(A) The high-boiling extraction media used according to E.P. 230,447 (cf. B., 1925, 827) to recover from the aqueous effluents the solvents (of higher b.p. than acetic acid) which remove the acetic acid from its superheated mixture with steam, are replaced by low-boiling chlorinated solvents, *e.g.*, trichloroethylene, chloroform, carbon tetrachloride, ethylene dichloride, etc. From the solutions obtained, the low-boiling solvent is distilled off, the high-boiling acetic acid extraction medium (*e.g.*, tar cresol) remaining. [B] A superheated mixture of acetic acid and water vapour is treated in an extraction column with a mixture of an acetic acid extraction medium (hydroxylic aromatic compounds, *e.g.*, tar cresol) and a low-boiling chlorinated solvent for the latter (*e.g.*, trichloroethylene). Mixed vapours of water, solvent, and a little extraction medium pass to a column in which a fractional condensation is effected, such that only a vapour mixture of solvent and a little water passes through a dephlegmator and condenser to a cold-washing column. The rest of the vapour entering the fractionating column, containing a mixture of nearly all the water, all the extraction medium, and a little solvent, condenses, and passes through another condenser to a separator, where a mixture of extraction medium and solvent falls to the bottom, and is returned to the extraction column, while practically pure water, containing a little extraction medium, rises to the surface, and is run off to the lower part of the washing column, where it meets the heavier mixture of solvent and a little water. Pure water issues from the top of the column, and from the bottom a mixture of solvent and extraction medium, which is returned to the tank.
B. FULLMAN.

Manufacture of organic compounds containing oxygen [methyl alcohol, etc.]. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 266,405, 19.10.25. Cf. E.P. 229,714; B., 1925, 338).—The catalytic production of methyl alcohol etc. from carbon oxides and hydrogen is carried out under pressure and at a high temperature with gas mixtures containing a high percentage of non-reacting gases; the process is circulatory, fresh gas mixture being added and treated gas removed to maintain a substantially constant composition of the circulating gas mixture. B. FULLMAN.

Manufacture of methyl alcohol and other oxygenated organic compounds. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 266,410, 23.10.25).—Coal gas, coke-oven gas, natural gas, cracking gas, etc., together with large quantities of water vapour and/or carbon dioxide, are partially oxidised catalytically (cf. E.P. 231,218; B., 1925, 357), the hydrocarbons being thereby converted into carbon monoxide and hydrogen. As catalysts, nickel-coated magnesium oxide or molten iron alloys are especially suitable. The product is passed either directly or after removal of sulphur compounds and/or adjusting the ratio of carbon monoxide to hydrogen if desired, over a suitable catalyst, with formation of methyl alcohol.
B. FULLMAN.

Production of dicyanodiamide. G. BARSKY, Assr. to AMERICAN CYANAMID CO. (U.S.P. 1,618,504, 22.2.27. Appl., 9.6.23).—The transformation of cyanamide into dicyanodiamide is controlled by maintaining a suitable hydrogen-ion concentration. The latter is then increased to prevent decomposition of the latter compound, which is recovered by evaporating the solution.
B. FULLMAN.

Manufacture of new complex metal alcoholates. CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING) (E.P. 261,377, 9.11.26. Conv., 12.11.25).—Complex mixed metallic alkoxides, such as the *aluminium magnesium ethoxides* $[Al(OEt)_4]_2Mg$ and $[Al(OEt)_6]_2Mg_3$, and *magnesium sodium* and *aluminium sodium ethoxide* are produced by heating the requisite alkoxides together,

preferably in the presence of a solvent, or by the action of a metallic alkoxide on another metal in the presence of alcohol, or by the action of alcohol on a mixture of two metals or an alloy, or by decomposing a metallic salt with excess of a metal alkoxide in alcoholic solution. These substances are very soluble in organic solvents, and are used as condensing agents. B. Fullman.

Process and apparatus for denicotining tobacco, tobacco waste, and tobacco products. J. Sartig (E.P. 265,427, 20.4.26).—The tobacco material is placed in a wire basket which rests on a perforated plate in a closed container. By means of an outer heating bath, the material is heated at below 100°. Into the container is run preheated water (if free nicotine is to be extracted) or water containing 0·25% of ammonia (for combined nicotine), air being excluded throughout the process. By continued heating, aqueous or ammoniacal vapours, after passing through the tobacco, impinge with their nicotine content on a sloping or conical cover, and are condensed, the liquid running down the sides without wetting the tobacco. B. Fullman.

Production of aryl-peri acids [phenyl-α-naphthylamine-8-sulphonic acid]. H. D. Gibbs and E. L. Frederick, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,617,313, 8.2.27. Appl., 11.10.20. Renewed 24.6.26).—Naphthylamine-8-sulphonic acid (100 pts.), aniline (300 pts.), and water (50 pts.) are heated under pressure at 170° for 5 hrs. when pure phenyl-α-naphthylamine-8-sulphonic acid is obtained. T. S. Wheeler.

Synthetic perfume. C. S. Miner, Assr. to Quaker Oats Co. (U.S.P. 1,617,412, 15.2.27. Appl., 26.12.22).—Esters of pyromucic acid and aliphatic alcohols are of value as perfumes, flavouring agents, and the like. T. S. Wheeler.

Manufacture of quinine solutions suitable for subcutaneous injections. F. B. Dehn. From Chem.-Pharm. A.-G. Bad Homburg (E.P. 266,209, 1.7.25).—Stable solutions of quinine are prepared by dissolving quinine in olive oil in the presence of a terpene, *e.g.*, terpineol, or a mixture of terpenes. B. Fullman.

Production of double salts of calcium halides with calcium lactate. J. Colman (E.P. 266,607, 28.7.26).—Non-deliquescent calcium halide-lactates, suitable for therapeutic use, are prepared by the action of 10% halogen hydracids on calcium lactate. The following are described:

$CaCl_2,2Ca(C_3H_5O_3)_2,CaH_2(C_3H_5O_3)_4$;
$CaBr_2,6Ca(C_3H_5O_3)_2,CaH_2(C_3H_5O_3)_4$, and
$CaI_2,10Ca(C_3H_5O_3)_2,CaH_2(C_3H_5O_3)_4$.

B. Fullman.

Pharmaceutical products. H. Hahl, Assr. to Winthrop Chemical Co., Inc. (U.S.P. 1,616,365—6, 1.2.27. Appl., [a], 5.10.25; [b], 16.12.25. Conv., [a], 28.11.24; [b], 19.2.25.—(a) A solution of the sodium salt of a hydroxyquinolinesulphonic acid is treated with excess of antimony hydroxide, the solution obtained is filtered, and treated with alcohol, when a *compound* of the type $[C_9H_{8-x}N(SO_3H)_xO]_3Sb$ is obtained. (b) Thioglycollic acid (20 pts.), antimony pentoxide (11 pts.), and water (500 pts.) are heated until complete solution occurs, the liquid is neutralised with sodium carbonate, and evaporated at a low temperature. A *product*, $[C_2H_4(CO_2Na)S]_5Sb$, is obtained. These products have therapeutic value. T. S. Wheeler.

Secondary butylallylbarbituric acid. A. W. Dox, Assr. to Parke, Davis and Co. (U.S.P. 1,615,870, 1.2.27. Appl., 7.8.23).—5-*sec.*-Butyl-5-allylbarbituric acid (cf. Volwiler, A, 1925, i, 1173) is claimed as a new compound of therapeutic value as a sedative. T. S. Wheeler.

Production of modified pneumococcic antigen and antipneumococcic serum. W. P. Larson (U.S.P. 1,621,117, 15.3.27. Appl., 27.8.25. Renewed 7.1.27).

Production of modified bacteria and toxins, and immunising serums. W. P. Larson (U.S.P. 1,621,118, 15.3.27. Appl., 4.11.25. Renewed 30.7.26).

Scarlet fever antigen. W. P. Larson (U.S.P. 1,621,119, 15.3.27. Appl., 27.1.26. Renewed 26.11.26).

Manufacture of diaminodiarylcarbamides. I. G. Farbenind. A.-G., Assees. of F. Heinze (U.S.P. 1,617,847, 15.2.27. Appl., 27.5.26. Conv., 10.6.25).—See E.P. 254,667; B., 1926, 769.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Contrast of photographic printing paper. L. A. Jones (J. Franklin Inst., 1926, **202**, 589—625; 1927, **203**, 111—156. Cf. B., 1926, 996).—Analysis of the errors involved in the sensitometry of printing papers shows that the reading error (involved in the measurement of density) is of the greatest magnitude, although of itself small. The total density error of a characteristic curve, based on the average of five sensitometric strips, should not exceed ± 0·015. The effect of time of development on the characteristic curve of papers is considered. With the exception of bromide papers, development is very rapid. Maximum density and maximum gamma (and contrast) are obtained relatively quickly, after which prolongation of development produces only a lateral shift of the entire curve. Contrast is dependent on development time unless development is carried to the point at which equilibrium in the shape of the curve is reached. Results of the sensitometric examination of each of the thirty-seven positive materials used are given. Four critical points are denoted on the characteristic curves. From the co-ordinate values of these points all of the necessary sensitometric constants, with the exception of D max., can be computed. This method gives additional sensitometric constants, and results in a more complete expression in numerical terms of the characteristics of the material as expressed by the D-log E function. The colour coefficient of the negatives is computed, using the sensitometric curve for each material, and the values of maximum and minimum density for the optimal prints. The method gives good agreement with direct determinations of colour coefficient by strictly sensitometric methods. Using the statistical data relating to the characteristics of the optimal positives, and the sensitometric data from the characteristic curves for the positive materials, the portion of the curve used in making each optimal positive is determined. The limiting gradient of the curves is discussed (*i.e.*, the value of dD/d log E at the point on the characteristic

curve which marks the limit of the utilised portion). The limiting gradient is not constant, but depends on the contrast of the positive material. There is no reason to believe that the limiting gradient in the high-light region is different from that in the shadow region for any particular optimal positive. The ratio of the shadow and high-light limiting gradients depends vitally on the printing exposure used in making the positive. A change in the printing time to equalise limiting gradients results in an improvement of photographic quality. The equalised limiting gradients for each of the optimal positives is determined. The following formula is evolved, for computing contrast in absolute terms: $\Omega = 1/(l\Delta D)^2.DS.\bar{G}(D)$, where $l\Delta D$ = least perceptible difference in density, = 0·007; DS = density scale; $\bar{G}(D)$ = average gradient evaluated with respect to equal density increments. A graphic method for evaluating contrast directly in terms of an enclosed area is derived, and the method is applied to determine numerical values of total contrast for each of the positive materials, and values of partial contrast for each of the optimal positives. The relation between contrast and magnitude of density scale is shown graphically for typical cases. Experimental evidence shows that it is not possible to use directly the value of the gradient evaluated with respect to equal increments in log E, for the evaluation of contrast. A contrast scale for practical use is proposed. W. Clark.

Mordanting of silver images by cupric thiocyanate. A. and L. Lumière and A. Seyewetz (Bull. Soc. Franç. Phot., 1927, **68**, 305).—See B., 1926, 933.

XXII.—EXPLOSIVES; MATCHES.

Mayrhofer's method for testing the stability of nitroglycerin powders. M. Tonegutti (Annali Chim. Appl., 1927, **17**, 60—65).—The test proposed by Mayrhofer (B., 1919, 603 A) and approved by Will (B., 1919, 926 A) does not allow of the detection of differences in the degree of stability of powders of the cordite type. The test does reveal differences in stability with powders of the ballistite type, but, owing mainly to the temperature employed, which is too high for powders so rich in nitroglycerin and devoid of added stability agents, its indications are not so distinct or certain as those furnished by the silvered vessel test or the Taliani test (cf. B., 1926, 174). T. H. Pope.

Patent.

[Sensitised] ammonium nitrate explosive. W. O. Snelling, Assr. to Trojan Powder Co. (U.S.P. 1,617,182, 8.2.27. Appl., 10.6.22; cf. 1,510,348; B., 1925, 28).—A saturated aqueous solution of ammonium nitrate (95 pts.) in water is mixed in the warm with a saturated, alcoholic solution of trinitrotoluene (5 pts.), and the mixture cooled, when ammonium nitrate with trinitrotoluene uniformly disseminated through it is obtained. Modifications of the process are described. T. S. Wheeler.

XXIII.—SANITATION; WATER PURIFICATION.

Calcium and magnesium hydroxides as coagulating agents. M. E. Flentje (J. Amer. Water Works Assoc., 1927, **17**, 253—260).—The water supply of Oklahoma City after preliminary sedimentation is aerated, softened with lime, clarified with iron and alum, settled, carbonated with flue gases, filtered, and chlorinated. The water contains very little non-carbonate hardness, and requires only lime for softening. Formerly just sufficient lime was added to the water to reduce the total alkalinity to 50 p.p.m., at which point little or no removal of magnesium took place. Enough lime is now added to give an excess of lime of 6—12 p.p.m. Over-treatment in this manner results in the precipitation of a part of the magnesium and a substantial saving in coagulants (iron and alum). Distilled water rendered turbid by addition of clay deposited from the raw water on addition of lime alone remains opalescent, but is clarified by treatment with lime and magnesium chloride. W. T. Lockett.

Critical review of the methods of analysing water, sewage, and effluents, with suggestions for their improvements. J. W. H. Johnson (Analyst, 1927, **52**, 128—142).—Methods now in use for water and sewage analysis are unsatisfactory owing to marked discrepancy between analytical results and practical requirements; inadequacy; inherent difficulties such as increase of nitrification products in modern purification processes; obscurity of biochemical conditions present in biological oxygen-absorption tests; and consequent uncertainty of results. Modifications of the fundamental organic nitrogen and oxygen-absorption tests are suggested: (1) A modified Kjeldahl process in which nitrites or nitrates do not interfere; (2) an albuminoid nitrogen process carried out under strictly controlled conditions, and with results in relatively close agreement with practical requirements; all acid oxidation processes are condemned. (3) Modification of the Royal Commission test which gives definite results strictly comparable *inter se.* The results so far recorded of this test are largely the outcome of unnatural conditions, and curves obtained should be strictly rectilinear. D. G. Hewer.

Dissolution of lead by water in pipes. A Farine (Schweiz. Chem.-Ztg., 1927, 29—32).—Under similar conditions, distilled water saturated with air, distilled water containing (*a*) air and carbon dioxide, (*b*) air and sodium bicarbonate, (*c*) air, sodium bicarbonate, and free carbon dioxide, when passed at a fixed rate through a tube packed with lead shavings, dissolved, respectively, 110, 10·5, 0·6, and 1·0 mg. of lead per litre. It follows that sodium bicarbonate exerts a strong protective action which is less strong in the presence of free carbonic acid. The results may be explained by the use of physico-chemical considerations, which indicate that in the presence of insoluble lead carbonate the concentration of the lead dissolved is directly proportional to that of the carbonic acid and inversely proportional to the square of the concentration of the bicarbonate. W. T. Lockett.

Patent.

Sterilising or pasteurising putrescible liquids. H. Stassano (E.P. 254,725, 30.6.26. Conv., 1.7.25).—The liquid, *e.g.*, milk, beer, etc., is allowed to flow at a rate of about 2 m./sec. through an annular space of a thickness of about 1 mm., formed by co-axially disposed metal tubes which are maintained at the required temperatures for pasteurisation or sterilisation, and adapted to cause the liquid to travel over a long path. W. T. Lockett.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 13, 1927.

I.—GENERAL; PLANT; MACHINERY.

Heat transfer alinement charts. M. Roulleux (Chem. Met. Eng., 1927, **34**, 148—151).—The practical solution of most problems relating to the transfer of heat between two fluids in circulation and separated by a solid wall involves the determination of the mean temperature difference. Curves have been derived mathematically by means of which this factor may be easily deduced. C. A. King.

Compressed gas handling technique. F. A. Ernst (Chem. Met. Eng., 1927, **34**, 151—153).—Attempts to adapt hydraulic and compressed air equipment to use with compressed gases has in most cases proved uneconomical. Several types of high-pressure valves and unions designed for this special use are described. Alloy steel tubing, although of higher unit price, is desirable by reason of the reduced weight of the fitting allowable. In a union fitting the gas pressure is held by a metal gasket, the threads serving merely to hold the tube ends against the gasket. For a closure for a reaction vessel, instead of an end plate being bolted to the flanged vessel with consequent distortion in use, the plate is fitted internally with a gasket, and the bolts are in compression. C. A. King.

Turbidimetry. Renwick.—See XXI.

Patents.

Cooling and drying or mechanically treating hot material which must not come in contact with the air until cold. N. V. Carbo-Union Ind. Maatschappij (G.P. 435,737, 4.12.24).—Activated charcoal, ultramarine, calcium cyanamide, and similar material is discharged direct into a chamber in which it is cooled by a current of a non-reacting gas which circulates through the chamber and through a heat-exchanger. Means are provided for collecting separately the coarse particles of the material and the fine particles carried along with the gas. L. A. Coles.

Heating of liquids. C. Callebaut and J. de Blicquy (E.P. 266,075, 4.12.25).—When a bath of liquid is heated by a steam pipe dipping into the bath, the vertical part of the pipe is liable to cause ebullition and splashing before the bulk of the liquid is sufficiently heated. To prevent this, the vertical part of the steam pipe is covered with a casing or sleeve, which may be either closed at the top and open at the bottom, or closed at the bottom and open at the top above the level of the liquid with the open end covered by a hood, or closed at both ends except for a vent pipe at the top, bent down over the bath of liquor. B. M. Venables.

Process of recovering volatile solvents [without explosions]. A. L. Mond. From Metallbank u. Metallurgische Ges. A.-G. (E.P. 266,145, 9.3.26).—In a process for recovering volatile solvents where a gaseous medium is continuously circulated through a drying or evaporating apparatus and a condenser or absorbing apparatus, explosions are prevented by using a mixture of oxygen and nitrogen containing less oxygen than air and/or by allowing the gases to take up solvent vapour until the upper explosion limit has been passed, and keeping above that point even in the condensing stage. To provide more internal heat for the evaporation a gas of high specific heat, *e.g.*, carbon dioxide, may be used in the mixture. B. M. Venables.

Machines for emulsifying, mixing, or grinding materials. R. Surtees (E.P. 266,098, 5.1.26).—In a colloid mill of the type wherein two discs rotate closely to each other at a high relative speed, each disc is formed with a groove on the working face, and fixed curved guides and collecting channels are arranged so as gradually to deflect and reduce the speed of the issuing emulsion. B. M. Venables.

Wet separation of constituents of mineral and other pulp. T. G. Martyn (E.P. 264,129, 30.8.26. Conv., 7.1.26).—Several forms of apparatus are described in which the pulp flows horizontally between confining surfaces which are a short distance apart, and so inclined that the pulp is continually meeting a surface after only flowing a short distance horizontally, and the solid matter when deposited in the nearly stationary film touching the surfaces slides along them to collecting means in the lower part of the apparatus. B. M. Venables.

Apparatus for the distillation or drying of coal or other solid materials. F. G. Hoffman (E.P. 261,740, 15.11.26. Conv., 17.11.25).—Through a cellular metallic structure comprising alternate rows of vertical and horizontal passages, the material passes downwards once only, and the heating gases zig-zag across. For continuous operation a number of cellular units may be arranged as the spokes of a wheel on a rotating carriage, the bottom being closed by an annular plate interrupted only at the discharging point. Products of distillation are collected by a surrounding casing, and the heating gases are supplied to and withdrawn from the central space. B. M. Venables.

Process and apparatus for spraying or projecting viscous, liquid, or granular material. K. Kisse (E.P. 262,066, 25.10.26. Conv., 30.11.25).—Material which may be of heterogeneous nature and liable to separate into its constituents, is delivered to the interior of a centrifugal bowl, up which it rises by centrifugal force, and is discharged over the top rim by a fixed scraper, which delivers it at a high speed in the required direction. The fixed scraper may be supported by the fixed central-feed hopper. B. M. Venables.

Apparatus for mixing liquids with solid or semi-solid substances or with other liquids. N. Bendixen, and Milkanic, Ltd. (E.P. 266,950, 25.5.26).—The device comprises a propeller and surrounding perforated screen rotating together in a vessel of the liquid to be emulsified. The rake of the propellers is such as to give a downward flow to the liquid, so that considerable pressure is created in the screen basket, and any entrained air will expand on emerging from the basket, thus aiding emulsification. A stationary guiding screen may be provided to circulate the liquid back to the basket. B. M. Venables.

Separation of the constituents of gaseous mixtures by liquefaction. L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude (E.P. 263,732, 17.3.26. Conv., 24.12.25).—A mixture of gases containing a wanted low-boiling constituent, *e.g.*, hydrogen, is compressed and cooled (1) by heat exchange with the returning unwanted liquefied gases, (2) by the hydrogen before expansion, and (3) by the hydrogen after expansion. The cooling stages (1) and (3) are separate, but (2) is effected simultaneously on the same portion of gas as (1) and (3). Stage (2) also effects reheating of the hydrogen prior to its doing work in the expanding machine. B. M. Venables.

Apparatus for separating constituents of gaseous mixtures. C. C. van Nuys, Assr. to Air Reduction Co., Inc. (U.S.P. 1,619,169, 1.3.27. Appl., 6.12.24; cf. U.S.P. 1,539,528; B., 1925, 575).—In the apparatus described, compressed cooled air is subjected successively to the cooling action of cold expanded gases and liquid products, and the liquid fraction resulting is rectified to produce a liquid as rich as possible in oxygen. This liquid is subsequently rectified, and the vapours produced are brought in contact with liquid nitrogen in quantity sufficient to ensure the separation of all the oxygen. T. S. Wheeler.

Apparatus for treating gases and vapours with silent electric discharges. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 266,983, 19.7.26).—Tubes of suitable size are placed loosely one inside the other, and are spaced at their respective upper ends by projections or pegs on the inner or outer tubes, and at the lower ends by constrictions on the outer tubes. J. S. G. Thomas.

Absorption refrigerating apparatus. P. M. Le Roy, Assce. of G. Beaumont (E.P. 255,053, 1.7.26. Conv., 8.7.25).—Ammonia vapour is conducted from the boiler absorber through a non-return valve to the ammonia liquefier, which is water-cooled and outside the space to be chilled. At the bottom of the liquefier are two valves, one leading to the evaporator or refrigerator proper, and the other for leading residual aqueous ammonia solution back to the boiler. The boiler absorber is also water-cooled during the absorption stage. B. M. Venables.

Refrigerating apparatus. J. O. Boving (E.P. 266,920, 26.3.26).—The condenser–evaporator and boiler–absorber of a refrigerator of the reversing absorption type are invertible, and are coupled together side by side as one unit by the transfer pipe, which is perforated within the boiler absorber and leads from one end of that vessel to the remote end of the condenser evaporator. All the necessary operations to complete a cycle of refrigeration are effected by tilting the apparatus to various positions. During the boiling off stage the boiler is heated and the absorber placed in a vessel of water. B. M. Venables.

Refrigerating and heat interchanging apparatus. D., M., S. R., and S. Guggenheim, J. K. MacGowan, and E. A. C. Smith, Assecs. of C. L. Burdick (E.P. 261,731, 5.11.26. Conv., 21.11.25).—A tubular heat interchanger is provided with devices for spreading the two liquids in a thin film on the interior and exterior of the tubes respectively. For the inner liquid, cones at the top, with their bases nearly touching the walls of the tubes, may be used, and for the exterior, one or more trays may be provided with holes slightly larger than the outside of the tubes. B. M. Venables.

Heat exchangers. Carborundum Co., Ltd. From Carborundum Co. (E.P. 266,100, 6.1.26).—A tubular heat exchanger is constructed of silicon carbide tubes which are good conductors, and are intended to be heated by radiation from the hot gases which may be "live" gases of combustion, and metal tubes which are used where the heat is not so great. All are supported and interconnected by a double metal shell, which is screened where necessary from excessive heat by refractory linings. B. M. Venables.

Heat exchange apparatus. Babcock & Wilcox, Ltd., A. Hall-Brown, and E. W. Jones (E.P. 266,206, 23.7.26).—A fan with alternate passages for gas and air is arranged at the cold end of a stationary, cellular heat interchanger. One fluid, say the gas, enters the eye of the fan and leaves the rotor tangentially, while the air enters through ports on the back plate and leaves in an axial direction, *i.e.*, the flows of gas and air are substantially at right angles. B. M. Venables.

Furnaces. Radiation, Ltd., S. N. Brayshaw, and E. R. Brayshaw (E.P. 266,487, 12.12.25).—In a regenerative gas furnace constructed as described in E.P. 119,553 (B., 1918, 678 A) those parts of the gas and air inlet pipes where the hot gases first strike them are covered by a loose, renewable piece of metal, and a deflector is placed in the combustion chamber in the path of the gas jet. B. M. Venables.

Filtering apparatus. R. Powley & Sons, Ltd., and M. Powley (E.P. 266,615, 28.8.26).—A vacuum drum filter is rotated by the incoming unfiltered liquid, and the vanes or buckets for this purpose may be on the drum itself. B. M. Venables.

Asbestos filter for the dry recovery of solid constituents of blast-furnace gases and the like. Deutsche Maschinenfabr. A.-G. (G.P. 435,653, 3.8.22).—The filter fabric has warp and weft constructed of tightly woven asbestos yarn, the warp and/or weft being wound round with loosely woven asbestos material. L. A. Coles.

Process and apparatus for obtaining powders of great fineness. E. Podszus (U.S.P. 1,621,270, 15.3.27. Appl., 2.9.24. Conv., 12.7.21).—See E.P. 183,134; B., 1923, 385.

Separation of constituents of gaseous mixtures containing hydrogen. G. Claude, Assr. to Lazote

Inc. (U.S.P. 1,620,192, 8.3.27. Appl., 12.3.23. Conv., 4.4.22).—See E.P. 195,950; B., 1924, 123.

Method of distillation [of tar etc.]. J. S. Morgan, Assr. to Thermal Industrial and Chemical (T.I.C.) Research Co., Ltd. (U.S.P. 1,622,964, 29.3.27. Appl., 30.1.23. Conv., 13.11.22).—See E.P. 207,366; B., 1924, 82.

Roof for industrial ovens and furnaces. O. Müller-Tanneck (E.P. 266,211, 20.8.26).

Roof for kilns or ovens. J. Laurent (E.P. 259,251, 4.10.26. Conv., 3.10.25. Addn. to 242,294).

[Fluid-cooled wall for] furnaces. T. E. Robertson. From Power Specialty Co. (E.P. 266,166, 10.4.26).

Checker work for regenerators of furnaces fired by gas producers. W. Lindner (E.P. 266,932, 21.4.26).

Irradiation apparatus for treating fluids with ultra-violet rays. Quarzlampen-Ges.m.b.H. (E.P. 247,231, 9.2.26. Conv., 9.2.25).

Heat insulating process, devices, and materials. E. Schmidt and E. Dyckerhoff (E.P. 266,177, 14.5.26).

Timing devices for centrifugal separators. E. A. Alliott and Manlove, Alliott & Co., Ltd. (E.P. 266,529, 10.2.26).

Liquid seals. A. H. Stevens. From Sharples Specialty Co. (E.P. 250,171, 19.11.25).

Apparatus for automatically governing the discharge of one liquid into another in proportion to the flow. R. Ames (E.P. 266,883, 4.2.26).

Means for heating by oil circulation. Manuf. de Machines Auxiliaires pour l'Electricité et l'Ind. (E.P. 262,753, 22.11.26. Conv., 10.12.25).

Vapour generating systems. Siemens-Schuckertwerke G.m.b.H., and W. Abendroth (E.P. 267,012, 10.9.26).

Drying cylinders. W. P. Evans (E.P. 266,481, 4.12.25).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Laboratory methods of determining the inflammability of coal dusts. A. L. Godbert (Safety in Mines Res. Bd., Paper No. 31, 68 pp.).—Previous work on the inflammation of coal dust is fully surveyed. The inflammability of a coal dust cloud is dependent on the concentration of the dust, its fineness, the nature of the coal, the presence of incombustible material, and the nature of the atmosphere in which the dust is suspended. Determination of the ignition temperature is unsatisfactory as a measure of the inflammability, the order of values obtained with a series of dusts being dependent on the method of measurement used. Moreover a dust of high ignition temperature may give rise to a more violent explosion than one of lower ignition temperature. The determination of speed of flame propagation presents experimental difficulties, particularly on a small scale. The most satisfactory laboratory method depends on the determination of the amount of incombustible dust which must be added to prevent inflammation. The results also are directly applicable to the problem of the prevention of explosions in coal mines. Details of the method which the Safety in Mines Research Board propose to adopt in their future experiments are briefly outlined. A. B. Manning.

Methods of analysis of coal. (Dept. Sci. Ind. Res., Fuel Res. Bd. Survey Paper No. 7, 35 pp.).—The methods of analysis recommended in the present report differ only slightly from those in the Interim Report (B., 1924, 206). The method of determining phosphorus in ash is slightly modified, and an appendix added dealing with possible errors arising from the presence of arsenic, silica, titanium, or vanadium. A somewhat more detailed account is given of the Gray-King carbonisation assay (B., 1921, 205 A). A. B. Manning.

Determination of nitrogen in fuels. G. Lambris (Brennstoff-Chem., 1927, **8**, 89—93; cf. B., 1927, 289).—Full details are given for carrying out the determination, and the accuracy and reliability of the method are exemplified by analyses of coals, coke, and various organic compounds. Two samples can be placed in the combustion tube at the same time, and their analysis carried out one after the other without any discontinuity. In this way two nitrogen determinations can be completed in 2—2½ hrs. W. T. K. Braunholtz.

Ten years of coal research. F. Fischer (Chim. et Ind., 1927, **17**, 367—374).—A brief account of the work of the Kaiser-Wilhelm Institut für Kohlen-Forschung at Mülheim-Ruhr.

Sulphur balance in coking practice. A. Weindel (Brennstoff-Chem., 1927, **8**, 85—89).—The quantity of sulphur present in raw coke-oven gas is calculated, and methods of recovering it, particularly the Burkheiser and Feld processes, are discussed. Whilst desulphurisation is essential for town's gas and gas for certain catalytic processes, the recovery of sulphur is nowadays less profitable than formerly, since gypsum is now largely used in the manufacture of ammonium sulphate and nitric acid is produced by oxidising ammonia. W. T. K. Braunholtz.

Removal of hydrogen sulphide from coke-oven gases. W. Gluud and R. Schönfelder (Stahl u. Eisen, 1927, **47**, 453—456).—A process is described, due to the Ges. für Kohlentechnik, in which the coke-oven gas immediately after the separation of the tar enters the bottom of a washing tower and meets a dilute suspension of ferric hydroxide in counter-current. The liquor containing ferrous sulphide is pumped into a tall tower called an "oxidiser." The sulphide liquor–air mixture rises into a settler where the sulphur is separated and the regenerated liquor passes again into the gas washer. The sulphur is floated out by the air into another vessel, where it is washed and dried. The air is led into an acid washer from which ammonium sulphate is drawn off from time to time. 85% of the sulphur in the gas is removed in the crude form, the balance in the solution being oxidised to sulphite and thiosulphate, the total recovery being as much as 99·5%. In order to prevent the solution getting too rich in salts, it is passed into the ammonia recovery apparatus, the iron lost being replaced by the addition of iron sulphate. Instead of iron sulphate or hydroxide, an alkaline soda solution may be used, which, although it obviates the

necessity of an acid washer for the air, is only recommended on special occasions. In conjunction with this installation a cyanogen washer was introduced, the cyanogen being recovered in the form of ammonium thiocyanate. T. H. BURNHAM.

Recovery of sulphur from gas. K. N. CUNDALL (Chem. Met. Eng., 1927, **34**, 142—147).—The removal of sulphur gases from fuel gas is based on a process devised by the Koppers Co., in which, however, the regeneration of the absorbing solution involved the emission of hydrogen sulphide into the atmosphere. Hydrogen sulphide is absorbed, according to the equation $Na_2CO_3+H_2S \rightleftharpoons NaHCO_3+NaHS$, by passing the gas through a tower filled with wooden hurdles over which a solution of sodium carbonate is sprayed. During experimental work on the interaction of sulphur dioxide and hydrogen sulphide in the presence of a catalyst, it was discovered that the oxidation of hydrogen sulphide to sulphur was completed in the presence of oxygen only, the most active catalyst being colloidal nickel sulphide formed from the addition of nickel sulphate to the foul solution. The process was therefore resolved into securing maximum contact between air and solution. Three types of aerators are in use. The "thioniser" type supplies air by means of canvas-covered perforated pipes placed at the bottom of a series of tanks through which the liquor flows. The Sirocco emulsifier is a superimposed set of four tanks each fitted with a turbine-driven, basket-type impeller. Very little power and no primary air are required for this installation. Large capacity is the feature of the Feld type emulsifier, a single 5 ft. 2¾ in. cone in a 13 ft. tank activating up to 100% efficiency at a rate of 1500 gals. of liquor per min. In all the plants sulphur separates as a grey muddy foam, which may form a layer several feet thick, and may then be filter-pressed and washed. The cake contains about 50% of water and 3% of ash, the recovery in the form of free sulphur averaging about 85%. The presence of cyanides reduces the efficiency of the catalyst. C. A. KING.

Flame temperature of producer gas with air and gas preheating. R. NITZSCHMANN (Chem.-Ztg., 1927, **51**, 197—198).—Equations are constructed based upon the composition of the producer gas, the volume of oxygen available for combustion, and the temperatures of the oxygen or air and producer gas. These are reduced to three constants, expressed in terms of the volumes of nitrogen and oxygen present before combustion, of carbon dioxide and water vapour after combustion, and of the specific heats and calorific values of reacting gases. The combination of these constants gives the theoretical flame temperature. A diagram is included for determining the flame temperature of producer gas of given composition when there is no preheating. H. D. GREENWOOD.

Gaseous explosions. IV. Rate of rise of pressure, velocity of flame travel, and the detonation wave. V. Probable mechanism causing "detonation" in the internal-combustion engine. G. G. BROWN and G. B. WATKINS (Ind. Eng. Chem., 1927, **19**, 363—369. Cf. B., 1927, 243).—When *iso*hexane–oxygen mixtures are exploded under conditions of constant initial temperature and pressure, the maximum rate of rise of pressure in the detonation wave occurs in mixtures containing slightly more oxygen than that just necessary to form carbon monoxide and hydrogen. A comparison of these results with those of Dixon on the explosion of ethylene–oxygen mixtures (Phil. Trans., 1893, **184A**, 97) and a consideration of other previous work indicates that the velocity of flame travel and the rate of rise of pressure in exploding mixtures are closely related, and vary in the same way with changing initial conditions. The amount of nitrogen which had to be added to various fuel–oxygen mixtures in order to reduce the intensity of detonation to an arbitrary standard varied directly as the rate of rise of pressure in the exploding mixture, which may therefore be taken as a measure of the tendency of the fuel to initiate detonation. The relative values of the rise of pressure for paraffin and aromatic hydrocarbons are entirely different from their relative tendency to produce "knocking" in internal-combustion engines; the cause of this phenomenon must therefore involve other factors than the detonation wave. A review of the available experimental evidence indicates that "knocking" in internal-combustion engines is caused by a heterogeneous reaction occurring at the heated cylinder walls, and not by the setting up of the detonation wave. The tendency of a fuel to cause knocking varies as the ratio of the rate of rise of pressure in an explosive mixture of the fuel to its auto-ignition temperature, which suggests that knocking is due to auto-ignition of the unburned mixture adiabatically compresssed against the hot walls of the cylinder. A. B. MANNING.

Combustion in the gasoline engine. I. Determination of rate of burning by chemical analysis. II. The burning of hydrogen and carbon monoxide. W. G. LOVELL and J. D. COLEMAN [with T. A. BOYD] (Ind. Eng. Chem., 1927, **19**, 373—378).—The composition of the gases in an engine cylinder has been determined at various times after ignition by means of a specially designed quick-acting, water-cooled sampling valve. The mixture of gases in the cylinder at any moment appears to be almost homogeneous. The data obtained from an air-cooled Delco-Light engine running at 1200 r.p.m. on gasoline with 75% of the theoretical air requirement have been plotted, and permit an estimate being made of the rate at which combustion is progressing in the cylinder. When "knocking" is induced by the use of a gasoline–kerosene mixture, or by the addition of a small quantity of *iso*propyl nitrite, the rate of burning is more rapid than normal, the normal rate being restored by the addition of lead tetraethyl. The gas composition–time curves obtained when the engine is driven on a mixture of equal volumes of hydrogen and carbon monoxide indicate that the simultaneous burning of these gases takes place thus:—$2H_2+O_2=2H_2O$; $2CO+O_2=2CO_2$, the ratio of the velocity constants $k_{H_2}:k_{CO}$ being 2·3. Neither alteration of the compression ratio nor the presence of ethyl nitrite or of lead tetraethyl alters the mechanism or the relative rates of these reactions. The results do not accord with the conception of gaseous combustion in which the flame is regarded as a boundary surface between burned and unburned gases, the gases in the cylinder at any instant being apparently homogeneous

and the reactions following the laws of homogeneous gas reactions. A. B. MANNING.

Laboratory method of determining the starting properties of motor fuels. W. G. LOVELL and J. D. COLEMAN [with T. A. BOYD] (Ind. Eng. Chem., 1927, 19, 389—394).—A measured amount of gasoline is charged into a bomb of 300 c.c. capacity, maintained at constant temperature, and the air–gasoline mixture vigorously agitated by means of a fan, the gasoline being contained in a small cup attached to the fan shaft. The bomb is provided with a spark gap, and after 2 min. agitation the fan is stopped and the ignition current turned on. The air–fuel ratio just necessary to produce an explosive mixture under these conditions is a measure of the ease of starting an engine when using the fuel, as is shown by a comparison with the results obtained directly in engine starting tests at the Bureau of Standards. Determinations have been made for a number of fuels at temperatures ranging from —15° to 20°. No direct relationship exists between the values so determined and the dew-points of the fuels, nor does it appear possible to obtain a quantitative measure of the readiness of starting from the Engler distillation curves. A. B. MANNING.

Improving fractionation in petroleum refining. D. B. KEYES (Chem. Met. Eng., 1927, 34, 164—165).—It is suggested that in the refining of petroleum by means of a bell-cap fractionating column, without reflux, connected to a pressure still by a vapour line and a liquid return, over-cracking may be prevented by the application of more modern systems of fractionation as used in other processes, *e.g.*, the distillation of alcohol. Owing to the formation of gummy substances during fractionation, the introduction of a number of storage plates in the fractionating column would be desirable in order to allow sufficient time for the deposition of gums. C. A. KING.

Solubility of naphthalene. RHODES and EISENHAUER.—See III.

PATENTS.

Coal washing jig. U. S. JAMES (E.P. 266,635, 21.10.26).—A jig is constructed so that the pulsations of water are upwards, alternating with periods of quiescence, there being no downward flow through the material under treatment. The pulsations are produced by pistons and suitable valves. B. M. VENABLES.

Heating of jacketed rotary retorts for the carbonisation of fuels at definite low temperatures. E. K. STACKMANN (G.P. 435,573, 1.1.21).—Retorts are heated by circulating hot gases through the surrounding jacket, and are maintained at the required temperature by the regulated addition of hot or cold gases to the circulating current. A. B. MANNING.

Recovery of constituents, other than sulphur dioxide, from gas mixtures. M. SCHROEDER (G.P. 435,587, 18.9.24).—In the recovery, *e.g.*, of benzene from coke-oven gas, of ethylene from illuminating gas, or of carbon dioxide from combustion gases, by absorption in a liquid under pressure, the potential energy of the compressed residual gases is utilised to generate power in a suitable engine, which energy is increased by heating the gas before it passes into the engine. L. A. COLES.

Apparatus for treating [cracking] hydrocarbons. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,618,645, 22.2.27. Appl., 15.7.20. Renewed 14.8.26; cf. U.S.P. 1,524,818; B., 1925, 276).—An apparatus for cracking hydrocarbons comprises a still in the form of a horizontal cylinder, the greater portion of which rotates around the axis. Heating is effected by combustion within a tube lying in the axis. All vapour outlets and other pipes are connected to the stationary portion of the cylinder. Thorough agitation of the liquid and uniform heating are obtained. T. S. WHEELER.

Method and apparatus for treating petroleum hydrocarbons. W. M. CROSS (U.S.P. 1,621,475, 15.3.27. Appl., 24.11.24).—In a continuous process for treating petroleum hydrocarbons the oil is intimately mixed with an acid for about ½ min. (to prevent polymerisation), and is separated mechanically. C. O. HARVEY.

Process of treating acid sludge to provide a fuel. W. D. KINKADE and C. J. BAUGH (U.S.P. 1,621,071, 15.3.27. Appl., 8.1.27).—Acid sludge from petroleum refining is added to a fuel oil, the mixture is heated to reduce the viscosity, and is agitated with amounts of soda and lime far less than those required for complete neutralisation of the sludge. C. O. HARVEY.

Apparatus for heating stills. G. D. WHITE, Assr. to TEXAS Co. (U.S.P. 1,621,298, 15.3.27. Appl., 5.4.23).—An apparatus for cracking hydrocarbon oils comprising a battery of spaced vertical stills surrounded by a furnace chamber, even distribution of heat being attained by (*a*) spacing the stills from the furnace walls, (*b*) avoiding direct contact between the flames and the still walls by a special distribution of the gas burners, which extend through opposite walls of the furnace, and (*c*) a series of flue passages. C. O. HARVEY.

Combined condenser and oil separator. F. C. BELL (U.S.P. 1,620,713, 15.3.27. Appl., 5.2.24).—The oil and gas are admitted to a cylindrical chamber containing an interior cylindrical casing carrying a spiral cooling coil. Condensation and separation occur, and the oil is drawn off from the base of the chamber by means of a pipe extending upwards into the oil strata. C. O. HARVEY.

Oil and gas separator. D. G. LORRAINE (U.S.P. 1,620,771, 15.3.27. Appl., 26.5.24).—A device comprising a settling chamber and a gas chamber, both fitted with valvular discharge controls operated automatically by means of a float. C. O. HARVEY.

Making grease. J. MCKEE, Assr. to SUN OIL Co. (U.S.P. 1,619,352, 1.3.27. Appl., 27.2.25).—Calcium hydroxide (100 pts.), mixed with a mineral lubricating oil (300 pts.), is heated at 130° to expel water, cooled to 38°, treated with oleic acid (600 pts.), mineral lubricating oil (4000 pts.), sodium hydroxide solution, *d* 1·21 (35 pts.), and a small quantity (1%) of water, and is finally heated with agitation at about 110°. T. S. WHEELER.

Manufacture of hydrocarbons and cyanides. J. C. CLANCY (E.P. 266,809, 2.12.25).—See U.S.P. 1,567,241; B., 1926, 486.

Apparatus for coal distillation (E.P. 261,740).—See I.

Asbestos filter for gases (G.P. 435,653).—See I.

Organic lead compounds (U.S.P. 1,622,228).—See XX.

Production of alcohols and hydrocarbons (F.P. 593,648—593,650, 594,121, and 598,447).—See XX.

III.—TAR AND TAR PRODUCTS.

Solubility of naphthalene in certain aromatic hydrocarbons. F. H. RHODES and F. S. EISENHAUER (Ind. Eng. Chem., 1927, **19**, 414—416).—The solubility of naphthalene in toluene, xylene, refined heavy solvent naphtha, and refined coal-tar creosote oil has been determined. For temperatures over a range of 0° to 50° the solubility curves for toluene, xylene, and solvent naphtha are roughly parallel, and the solubility at any given temperature decreases as the mol. wt. of the solvent increases. The addition of cresol has no specific effect in increasing the solubility of naphthalene in solvent naphtha or creosote oil. E. H. SHARPLES.

PATENTS.

Preparation of pure *p*-xylene. E. MERCK, Assr. to A. DÜTZMANN (G.P. 434,988, 27.9.24).—*p*-Methylbenzyl chloride, b.p. 80°/8 mm., prepared, for example, by condensing toluene with trioxymethylene and hydrogen chloride at 60° in the presence of zinc chloride, is reduced to pure *p*-xylene. A quantitative yield is obtained with palladium and hydrogen, rather less with zinc dust or aluminium amalgam. C. HOLLINS.

Distillation of tar (U.S.P. 1,622,964).—See I.

IV.—DYESTUFFS AND INTERMEDIATES.

Naphthalenesulphonic acids. VII. Hydrolysis of naphthalene-1 : 6-disulphonic acid. J. A. AMBLER and J. T. SCANLAN (Ind. Eng. Chem., 1927, **19**, 417—420).—Quantitative examinations have been made of the degree of hydrolysis occurring when pure naphthalene-1 : 6-disulphonic acid is heated in sealed tubes with concentrations of sulphuric acid ranging from 1% to 85% and at temperatures varying from 100° to 230°. The temperatures of the commencement of hydrolysis for each concentration of sulphuric acid and the nature of the reaction products have been determined. The following general rule has been deduced. Naphthalene-1 : 6-disulphonic acid, when heated with sulphuric acid of a concentration not high enough to sulphonate naphthalene at the temperature employed, and at a temperature sufficiently high to effect hydrolysis, is converted directly into naphthalene. If the temperature is high enough to hydrolyse the 1 : 6-acid and the sulphuric acid strong enough to sulphonate naphthalene at the temperature used, isomeric disulphonic acids, *i.e.*, those which would be produced by the interaction of naphthalene and sulphuric acid under the same conditions of temperature and concentration, are formed. During hydrolysis both sulphonic acid groups of the 1 : 6-acid are removed simultaneously, and there is no intermediate formation of the β-acid. On heating the 1 : 6-acid at temperatures at which hydrolysis begins in an open system, the reaction will go to completion if provision is made for the removal of the naphthalene, and thus, in the vapour phase sulphonation of naphthalene, which is carried out in an open system at 220—245° in 80—95% sulphuric acid, the 1 : 6-acid will not be present in the product. Also the 1 : 6-acid would not be affected in the steam treatment for the removal of tar, which is conducted at 100° in 10% sulphuric acid. (Cf. B., 1925, 66, 310.) E. H. SHARPLES.

Behaviour of oxides of lead towards dyestuffs. I. A. N. ADAMSON and J. K. WOOD (J. Soc. Dyers and Col., 1927, **43**, 47—51).—The absorption of the acid dyes Disulphine Green I, Patent Blue, Methyl Orange, Bordeaux B, Orange II, and Orange IV (the basic dyes Methylene Blue, Toluidine Blue, Safranine T, and Methyl Violet 10 B were not absorbed) by various samples of impure lead dioxide containing 92·6—98·5% PbO_2, 1·0—5·8% PbO, and 0·4—1·2% of moisture appears to be a case of adsorption. Less dye (Orange II or Methyl Orange) is sorbed in the presence of sodium hydroxide, the reduction in the degree of sorption being independent of the amount of alkali present, whereas the presence of an acid increases the sorption. Dyes adsorbed by the samples were not completely removed by washing with water. It is probable that lead dioxide free from lead monoxide would have but little power of adsorption. The complete inability of lead dioxide to adsorb basic dyes is in striking contrast to the behaviour of the oxides of titanium and tin (Morley and Wood; B., 1923, 544 A, 545 A), and is probably to be associated with the definite basic properties of lead dioxide and its low degree of hydration. A. J. HALL.

PATENTS.

Triarylmethane dyes. I. G. FARBENIND. A.-G., Assees. of A.-G. F. ANILIN-FABR. (E.P. 249,160, 13.3.26. Conv., 14.3.25; and E.P. 250,576, 1.4.26. Conv., 9.4.25. Addn. to E.P. 249,160).—Basic violet dyes of the Victoria Blue type, which may be converted into acid dyes by sulphonation, are obtained by condensing either 1 or 2 mols. of a tetra-alkyl-4 : 4′-diaminobenzophenone with 1 mol. of an *NN′*-diarylated ethylenediamine in the presence of phosphoryl chloride. Michler's ketone is thus condensed with *NN′*-diphenyl-*NN′*-dimethyl- (or diethyl- or dibenzyl-) ethylenediamine, *NN′*-di-*o*-tolylethylenediamine, and *NN′*-di-α-naphthylethylenediamine. C. HOLLINS.

Manufacture of sulphuric acid esters of oxyalkyl compounds of the aromatic series [sulphatoalkyl ethers of phenols]. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 266,940, 4.5.26).—Intermediates, suitable for manufacture of soluble dyes particularly for acetate silk, are prepared by the action of sulphating agents (sulphuric acid, chlorosulphonic acid, etc.) on hydroxyalkyl ethers of phenols, naphthols, anthrols, etc. *3-Nitro-p-tolyl β-hydroxyethyl ether*, m.p. 40°, obtained from 3-nitro-*p*-cresol and ethylene chlorohydrin, treated with concentrated sulphuric acid, yields a sulphate isolable as the *potassium* salt, $NO_2 \cdot C_6H_3Me \cdot O \cdot CH_2 \cdot CH_2 \cdot O \cdot SO_3K$, which may be reduced, diazotised, and coupled with β-naphthol for a soluble bluish-red dye, or the reduced nitro-compound may be coupled with *p*-nitrodiazobenzene for a soluble yellowish-red dye suitable for acetate silk. *Ethylene glycol mono-α-anthracyl ether*, m.p. 117—118°, and 2-β-hydroxyethoxynaphthalene also form sulphates.

From 1-βγ-dihydroxypropoxynaphthalene the monosulphate, $C_{10}H_7 \cdot O \cdot CH_2 \cdot CH(OH) \cdot CH_2 \cdot O \cdot SO_3Na$, is obtained. C. Hollins.

Manufacture of coeruleinsulphonic acids. I. G. Farbenind. A.-G., Assees. of A.-G. f. Anilin-Fabr. (E.P. 251,968, 26.4.26. Conv., 7.5.25).—Coeruleins, *e.g.*, from pyrogallolphthalein or from hydroxyquinolphthalein, are converted into soluble sulphonic acids, suitable for mordant dyeing, by treatment with fuming sulphuric acid and boric acid at 130—140°. C. Hollins.

Manufacture of [perylene] dyestuffs. A. Zinke (E.P. 242,306, 30.10.25. Conv., 3.11.24).—Halogenated diaroylperylenes, in particular 3 : 9-dibromo-4 : 10-dibenzoylperylene (cf. A., 1925, i, 383), are treated at high temperatures with alkalis or alkaline-earths in the presence or absence of organic solvents, in particular aniline. The products dye cotton from a blue vat in violet shades fast to chlorine, acids, alkalis, and washing. A. Davidson.

Copper [compounds of] azo dyestuffs. T. H. Leaming, Assr. to National Aniline & Chemical Co., Inc. (U.S.P. 1,618,762, 22.2.27. Appl., 22.6.25).—A solution of copper sulphate or other soluble copper salt is added to a neutral or slightly alkaline aqueous solution of an azo dyestuff obtained by coupling, in presence of sodium carbonate, 1 or 2 mols. of a diazotised anthranilic acid, or 1 mol. of the latter and 1 mol. of another diazo-compound, with 1 mol. of 5 : 5′-dihydroxy-2 : 2′-dinaphthylamine-7 : 7′-disulphonic acid. The mixture is made alkaline, and the copper derivative of the dyestuff formed is salted out. These products dye cotton and silk bright reddish-violet shades. T. S. Wheeler.

Manufacture of [azo] dyestuffs. F. Straub and H. Schneider, Assrs. to Soc. of Chem. Ind. in Basle (U.S.P. 1,617,872, 15.2.27. Appl., 11.3.26. Conv., 28.3.25; cf. E.P. 249,884).—Azo dyes, derived from *o*-phenolazo-2-α-naphthol-8-sulphonic acid in which positions 3, 4, and 6 of the naphthalene nucleus are substituted at least once, and at most twice, by non-adjacent sulpho-groups, of which one is in the 3- or 4-position, are treated with a metallic, *e.g.*, copper or chromium, salt to yield *dyes*, which give on wool in an acid bath fast, pure, bordeaux to violet, blue, and black shades. *E.g.*, diazotised *p*-chloro-*o*-aminophenol is coupled with α-naphthol-3 : 8-disulphonic acid, and an aqueous solution of the product is treated with copper sulphate and sodium acetate to give a substance which dyes wool red-violet shades. T. S. Wheeler.

[Mordant] azo dyes. British Dyestuffs Corp., Ltd., K. H. Saunders, and H. Goodwin (E.P. 266,561, 17.4.26. Addn. to E.P. 242,061; B., 1926, 7).—1-Amino-2-hydroxy-6-sulpho-3-naphthoic acid is diazotised and coupled with a sulphonated naphthol or dihydroxynaphthalene, to give reddish- to greenish-blue chrome printing colours for cotton or mordant dyes for wool. Examples of end-components are α-naphthol-4 : 8-disulphonic acid, 1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid, and 1 : 8-dihydroxynaphthalene-4-sulphonic acid. C. Hollins.

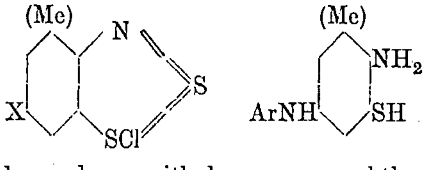
Disazo dye of diphenylcarbamide and chromotrope acid. R. J. Fletcher, Assr. to Amalgamated Dyestuff and Chemical Works, Inc. (U.S.P. 1,617,244, 8.2.27. Appl., 19.4.26).—Diazotised *p*-nitroaniline is coupled with 1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid, and the product is treated with sodium sulphide to reduce the nitro-group and then with phosgene to give s-*di*-(1 : 8-*dihydroxy*-3 : 6-*disulphonaphthalene*-2-*azo*-p-*phenyl*)*carbamide*, which gives direct brilliant violet shades, fast to light, but soluble in water. The compound can also be prepared by coupling diazotised *p*-aminoacetanilide with 1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid, hydrolysing the acetamido-group, and treating the product with phosgene, or by coupling tetrazotised *pp*′-diaminodiphenylcarbamide with 1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid. T. S. Wheeler.

Manufacture of azo dyes. W. Carpmael. From Farbenfabr. vorm. F. Bayer & Co. (E.P. 266,771, 5.10.25).—Arylsulphamic acids coupling in the *para* position are used as middle components in the manufacture of secondary disazo pigment dyes or ice-colours. Examples are: *o*-anisidine→α-naphthylsulphamic acid →2 : 3-hydroxynaphthoic anilide or β-naphthylamide (black on the fibre) or 1-phenyl-3-methylpyrazolone (scarlet on the fibre); aniline→bisacetoacetyltolidine (orange on the fibre). [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 238,683 (B., 1926, 840).] C. Hollins.

Manufacture of monoazo dyes. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 265,767, 30.1.26).—Toluidine-ω-sulphonic acids are diazotised and coupled with non-sulphonated, non-carboxylated arylamines, and nitrotoluidinesulphonic acids are diazotised and coupled with β-naphthylamine derivatives, including aminonaphthols and carboxylic and sulphonic acids. The products dye acetate silk in shades fast to washing, and unmordanted wool in level shades fast to light. 23 examples are given, including the coupling of diazotised *p*-toluidine-ω-sulphonic acid with diphenylamine (yellow) and methyl-β-naphthylamine (orange); 5-nitro-*o*-toluidine-ω-sulphonic acid with benzylethylaniline (red), methyldiphenylamine (red), 2-β-hydroxyethylnaphthylamine (red-violet), 2-methylaminonaphthalene-7-sulphonic acid (red-violet), γ-acid (violet), and methyl-γ-acid (blue-violet); 3 : 5-dinitro-*o*-toluidine-ω-sulphonic acid (G.P. 141,783) with ethyl-β-naphthylamine (blue-violet), methyl-γ-acid (blue), 2 : 3-aminonaphthoic acid (red-violet), 2 : 8-aminonaphthol (blue-black), Brönner's acid (bordeaux), phenyl-Brönner's acid (violet); and 3 : 5-dinitro-*p*-toludine-ω-sulphonic acid (G.P. 134,988) with β-naphthylamine (blue-red), ethyl-β-naphthylamine (bordeaux), and 2-methylamino- or 2-ethylaminonaphthalene-7-sulphonic acid (bordeaux). Monoazo mordant dyes (cf. E.P. 18,139 of 1902) are disclaimed. C. Hollins.

Manufacture of new aromatic compounds and dyes therefrom. A. J. Ransford. From L. Cassella & Co., G.m.b.H. (E.P. 265,641, 7.8.25).—Compounds of the type (I), prepared from arylamines and sulphur chloride (E.P. 17,417/14), where X is a labile substituent such as halogen or alkoxyl, react with arylamines to form

arylamino-derivatives, which are converted by alkalis, especially in the presence of a reducing agent, into 2-amino-5-arylaminothiophenols (II). The latter

(I)  (II)

compounds condense with benzo- or naphtha-quinones to give violet to blue to black quinone vat dyes for wool, which probably contain the thiazine nucleus. Examples are given of the preparation of 5-anilino-3-mercapto-*o*-toluidine (II, Ar = Ph) and its condensation with chloranil for a violet-blue dye; of 1-amino-4-anilino-2-thionaphthol and its condensation with 2-hydroxy-1 : 4-naphthaquinone or 2 : 3-dichloro-1 : 4-naphthaquinone for grey-blue to black dyes; of 5-anilino-3-mercapto-*o*-anisidine and its condensation with chloranil for an indigo-blue dye; of 5-(*o*- and *m*-carboxyanilino)-3-mercapto-*o*-toluidines and their condensation with chloroanil etc. for violet to greenish-blue dyes. Alternative routes to the dyes and intermediates are also covered. C. HOLLINS.

Preparation of 2 : 7-dintroanthraquinone. GRASSELLI DYESTUFF CORP., Assees. of B. STEIN (U.S.P. 1,622,168, 22.3.27. Appl., 28.6.26. Conv., 25.6.25).—Tetranitrodianthrone is treated with pyridine to form pyridine salts of tetranitrodianthranol, which is converted into 2 : 7-dinitroanthraquinone by removal of pyridine with acid followed by oxidation with concentrated nitric acid. C. HOLLINS.

Manufacture of alanines of the anthraquinone series and derivatives thereof. I. G. FARBENIND. A.-G., Assees. of FARBENFABR. VORM. F. BAYER & Co. (E.P. 246,840, 26.1.26. Conv., 31.1.25).—β-Chloropropionic acid is condensed with aminoanthraquinones, or β-alanine with halogenated anthraquinones, giving β-alanines of the anthraquinone series. Examples are the condensation of β-chloropropionic acid with 2-amino-, 1 : 4-diamino-, and 1 : 4 : 5 : 8-tetra-amino-anthraquinones. The products dye cellulose acetate and animal fibres. C. HOLLINS.

Manufacture of vat colouring matters of the anthraquinone series [acylated aminoanthraquinones]. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 264,561, 10.10.25).—Aminoanthraquinones are acylated in at least one α-amino-group by benzoic acid derivatives substituted in the *m*-position by a difficultly attackable group such as alkyl, alkoxy, etc. The acylation is accomplished by boiling the amine in a suitable solvent with the *m*-substituted benzoyl chloride or benzoic anhydride, or by heating a mixture of the amine and the *m*-substituted benzoic acid in the presence of a condensing agent such as thionyl chloride, phosphorus pentachloride, etc. The examples given include, as acylating agents, *m*-methoxy- and -ethoxy-benzoic, *m*-toluic, *iso*phthalic, *m*-benzamidobenzoic, 3-methylthiolbenzoic, diphenyl-*m* : *m'*-dicarboxylic, trimesic, and 5-methoxy*iso*phthalic acids, whilst the amines used include 1-aminoanthraquinone, 1 : 4- and 1 : 5-diaminoanthraquinones and their monobenzoyl derivatives, 1 : 5-diamino-4-hydroxy- and -4-methoxy-anthraquinones, and 4 : 8-diaminoanthrarufin. 1-*Amino*-5-*methoxyanthraquinone*, m.p. 225°, obtained from 5-chloro-1-aminoanthraquinone and sodium methoxide, is also used. The products are vat *dyes* of good affinity and fastness to light. A. DAVIDSON.

Manufacture of vat dyestuffs [dialkoxy*iso*dibenzanthrones]. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 264,631, 19.11.25).—Alkyl ethers of hydroxybenzanthrones substituted by halogen in the 3-position, but with a free 4-position, are treated with alkaline condensing agents. *E.g.*, 3-chloro-2-methoxybenzanthrone (obtained, *e.g.*, by treating 2-methoxybenzanthrone with sulphuryl chloride in nitrobenzene solution or by methylating 3-chloro-2-hydroxybenzanthrone) is heated with potassium ethoxide for 1 hr. at 145—150°, diluted with water, boiled, and aerated, when the dye is precipitated. It gives a blue vat, and dyes cotton greenish-blue. Excluded from the patent are those halogenoalkoxybenzanthrones obtained by nitrating 3-chlorobenzanthrone, reducing the nitro-compound, substituting a hydroxyl for the amino-group, and alkylating the product. A. DAVIDSON.

Manufacture of condensation products of the anthracene series [*Bz*-methylbenzanthrones]. O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS, UND BRÜNING (E.P. 244,120, 4.12.25).—Anthrones or anthranols are condensed with crotonaldehyde in the presence of pyridine and piperidine and subsequently cyclised to *Bz*-methylbenzanthrones by means of aluminium chloride. By using an acid condensing agent (hydrogen chloride, sulphuric–acetic acid) in place of the base the reaction proceeds to completion in one stage. An oxidant (arsenic acid) may be added. 1-(or 3-)Methylbenzanthrone, m.p. 113—114°, from anthranols and crotonaldehyde in sulphuric–acetic acid at 115—120°, distils in steam superheated at 300°. By the two-stage process anthrone and crotonaldehyde give a *Bz*-methylbenzanthrone, m.p. 168°. From α-hydroxyanthranol a hydroxy-*Bz*-methylbenzanthrone is obtained. C. HOLLINS.

Colour lakes (E.P. 265,032).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Sizing of artificial silk. W. BRUCKHAUS (Kunstseide, 1926, 8, 319—320).—Pastes containing glue or gelatin are much more suitable than those containing ordinary starch for sizing artificial silk yarn so that it retains its lustre and acquires suitable elasticity for purposes of weaving. Suitable sizes are prepared by (1) boiling 1 kg. of bone-gelatin (previously steeped in cold water for 12—24 hrs.), 1 kg. of gelatin, and 24 litres of water, then adding 200 g. of olive oil, 50 g. of anhydrous sodium carbonate, and 100 g. of glycerin, or (2) boiling 2 kg. of glue (previously steeped in cold water for 12 hrs.) and 25 litres of water, then adding 500 g. of Marseilles soap and 150 g. of Brilliant Monopol Oil G; both sizing preparations are used at 50°. A. J. HALL.

Viscose solutions. II. Turbidity measurements of the changes of state. T. MUKOYAMA (Kolloid-Z., 1927, 41, 158—163).—The effects on the

turbidity of solutions of viscose due to changes of concentration, age, temperature, and alkali content were studied. The turbidity of a freshly prepared sol increases linearly with concentration, but after a few days the increase of turbidity with concentration becomes much greater, reaches a maximum, and then falls off, the departure from the original curve increasing with the age of the sol. The maximum values for middle concentrations correspond with the maximum viscosity, and in old sols the phenomena of gelatinisation and syneresis are observed. In preparations containing a high percentage of alkali the maximum is followed by a minimum, but this is due to removal of some of the substance by precipitation. With rise of temperature the turbidity diminishes. With increasing content of alkali the turbidity also diminishes, the effect being greater the older the sol. E. S. Hedges.

Viscosity of viscose solutions. P. Waentig (Kolloid-Z., 1927, **41**, 152—157).—Experiments are given which show that when cellulose is very finely powdered before conversion into viscose the product has a much smaller viscosity than ordinary viscose. Such finely-divided cellulose is also much more readily attacked by solutions of sodium hydroxide. The decrease in viscosity of solutions of viscose (cf. Heuser and Schuster; B., 1926, 399) with age is discussed in the light of these experiments. Such an ageing effect takes place only when the excess of caustic soda has been removed. E. S. Hedges.

Solubility of cellulose derivatives. W. von Neuenstein (Kolloid-Z., 1927, **41**, 183—185).—Ostwald's peptisation rule (A., 1927, 310) is found to hold for the dissolution of cellulose acetate in chloroform and in ethyl acetate, and for cellulose nitrate in a mixture of alcohol, benzene, and acetone, the amount dissolved increasing with the amount of colloid initially present. Cellulose nitrate after extraction gave a curve showing that the amount dissolved by the liquid mixture is, above a certain concentration, independent of the initial quantity present. E. S. Hedges.

Autoxidation of lignin. H. Ditz and R. May (J. pr. Chem., 1927, [ii], **115**, 201—215).—Fischer and Schrader (A., 1922, i. 637; 1923, i, 185) found that, in presence of sodium hydroxide solution, lignin absorbs oxygen more readily than does pine-wood sawdust, and suggested that the lignin in the latter has a protective covering of cellulose. Consideration of the lignin content of the two specimens indicates, however, that that of the pine-wood is actually the more readily oxidised. This is attributed to the presence of catalysts. Since manganese is an important inorganic constituent of pine-wood, its effect on the autoxidation of purified, manganese-free lignin suspended in $2N$-sodium hydroxide has been examined. Addition of 0·05% of bivalent manganese produces an initial increase in the rate of oxidation, which disappears after the first 24 hrs. With 0·5% of manganese the increase is not so marked. This effect is probably not purely catalytic, the oxidation of the lignin being induced by the simultaneous autoxidation of the manganese to the quadrivalent state (cf. Meyer and Nerlich; A., 1921, ii, 509). H. E. F. Notton.

Pulping flax straw. Hydrolysis with sodium sulphite. M. W. Bray and C. E. Peterson (Ind. Eng. Chem., 1927, **19**, 371—372).—The digestion of flax straw at 155° with 20% and 40% of sodium sulphite at a concentration of 40 g./litre and with 40% of sodium sulphite at concentrations of 40 and 80 g./litre has been studied. Whilst an increase in the percentage of sodium sulphite greatly accelerates the rate of cooking, an increase in the concentration of the cooking liquor has but little effect. During the first hour the incrustants are removed at a rapid rate, and it is during this period that the loss of cellulose by hydrolysis is most pronounced. Beyond this point the rate of change of yield and chemical properties with time is comparatively slow. Pentosans in the cellulose as obtained by Cross and Bevan's method remain practically constant during the whole cooking period. Details are given of the chemical properties of the pulps obtained in these experiments. D. J. Norman.

Pulping flax straw. II. Chemical studies with chlorine as a pulping agent. E. R. Schafer, M. W. Bray, and C. E. Peterson (Pulp and Paper Mag., 1927, **25**, 209—212).—The pulping of flax straw by digestion with soda followed by chlorination has been studied in order to determine the relationship between the proportion of soda used in the preliminary digestion and the quantity of chlorine required to complete the pulping. For soda pulps prepared by digestion for 2—12 hrs. at a maximum temperature of 155° and showing soda consumption of 2—18·7% (on the weight of straw), there is a linear relationship between the soda consumption and the chlorine requirements, the latter ranging from 23·9 to 2·7% on the weight of straw. The final pulps showed practically identical properties irrespective of the relative amounts of soda and chlorine used in their preparation. D. J. Norman.

Soda pulp investigation. II. Yield and quality of pulp obtained from the birches and maples. D. E. Cable, R. H. McKee, and R. H. Simmons (Pulp and Paper Mag., 1927, **25**, 243—247; cf. B., 1926, 1007).—The data of a series of experimental cooks on white birch (*Betula papyrifera*), yellow birch (*Betula latea*), silver maple (*Acer saccharinum*, L.), hard maple (*Acer saccharum*, Marsh), and red maple (*Acer rubrum*, L.) are given. The samples were digested with 25% of soda on the weight of dry chips at 170° for 4—7 hrs., 4—5 hrs. being sufficient for white birch, yellow birch, and silver maple. Variations in the causticity of the fresh cooking liquor from 60 to 98% had no apparent effect on the yield and character of the resulting soda pulps, provided that the other cooking conditions were unchanged. The yields of pulp varied from 43·3% for hard maple and red maple to 44—48·6% for the other woods; calculated on a cord basis, these figures are equivalent to 1620—1980 lb. of dry pulp per cord of wood. The bleach requirements of these pulps were 10·7—14·7% (on the weight of air-dry pulp). Pitting of digester covers, which is attributed to the presence of traces of volatile organic acids in the digester vapours, should not be more serious when cooking birch and maple than is at present the case with aspen. D. J. Norman.

Recovery and utilisation of waste liquors in the pulp industry. U. Pomilio (Ind. Eng. Chem., 1927,

19, 344—346).—In view of the fact that one of the main difficulties attending the economic treatment of the waste liquors from the cellulose industry is their low concentration, attention is drawn to the chlorination process for manufacturing pulp, in which, by continued re-use of the liquors, it is possible to obtain a final alkali waste liquor containing about 150 g./litre of organic matter. Hägglund's process (cf. E.P. 258,035; B., 1927, 165), which involves heating the liquor under pressure, is suggested as being the most promising method of treating these liquors and also those obtained in the soda process. D. J. NORMAN.

Waste sulphite liquor as an agricultural spray. C. S. FLEMING and J. H. REEDY (Chem. Met. Eng., 1927, **34**, 159).—For use as an agricultural spray, it is suggested that sulphite waste liquor be saturated with hydrogen sulphide, thereby reducing calcium hydrogen sulphite with the separation of free sulphur. The liquor contains 7—9% of a sugar-like substance which aids the suspension of the sulphur particles, the permanence of which is improved by the addition of 0·5% of gelatin or maize sugar. If the sulphur is flocculated and separated, the solution contains fermentable material which might be utilised for the production of alcohol. C. A. KING.

Degree of sizing and finish [of paper]. H. SCHWALBE (Papier-Fabr., 1927, **25**, 173—176).—A review of various methods for determining the degree of sizing of paper is given, that due to Albrecht being particularly described (Finska Pappers- och Trävarutidskr. för Finland, 1926, Nos. 10 and 11). With this apparatus reliable results may be obtained, and the method also enables the disposition of the size to be observed. The humidity of the air and temperature of the ink both influence the size testing, 65% R.H. and 24° being recommended as standard conditions. Curves for samples examined show that for a rise in temperature from 10° to 30° the time of penetration of a water-glass-sized wood-pulp paper decreased only slowly, but for rosin- and animal-sized rag papers a rapid decrease was shown. Concordant results are obtained for samples lying adjacent to one another in the same sheet. Variation in the experimental conditions (*e.g.*, increasing the depth of the ink column in penetration tests) affects the results. Differences due to the effects of moist- and dry-glazing actions of the paper machines and also of calendering are indicated for samples tested before and after these operations. B. P. RIDGE.

Spanish moss. SCHORGER.—See XX.

PATENTS.

Manufacture of artificial silk. BRITISH ENKA ARTIFICIAL SILK CO. LTD., Assees. of N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (E.P. 259,528, 18.11.25. Conv., 12.10.25).—Threads of artificial silk of different denier are kept apart during manufacture by dyeing the silk with different colours according to the denier, either during or after spinning, such colours being subsequently removed or decolorised. B. P. RIDGE.

Manufacture of artificial silk. C. C. JESSEN, Assr. to ATLAS POWDER CO. (U.S.P. 1,621,590, 22.3.27. Appl., 20.3.25).—The thread is wound first into a cake in a centrifuge and then from the outside of the cake on to a cylinder, the latter being of such size with respect to the diameter of the cake that, as the thread is wound upon it, those portions which were under the greatest tension in the centrifuge are subjected to the least tension during winding, and *vice versa*. B. P. RIDGE.

Manufacture of viscose solutions. S. NEUMANN (E.P. 265,685, 10.11.25).—Suitable liquids are emulsified with completely dissolved viscose in high-speed mixers running at speeds greater than 1000 r.p.m. This treatment homogenises the viscose solution, provides a means of introducing liquid paraffin and other hydrocarbons, and accelerates the ripening process to such an extent that by adjusting the duration of the mechanical treatment and the temperature, which, however, should not exceed 50—60°, it is possible to complete the ripening process in a few hours. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 234,039, 189,114, 184,533, 143,253, and 1020/98 (cf. B., 1925, 914; 1923, 179 A; 1922, 806 A; 1921, 689 A).] D. J. NORMAN.

Production of cellulosic material. A. H. LYMN and C. LEYST (E.P. 265,697, 14.11.25).—A process is described for preparing crude bamboo pulp, which, when formed into blocks and dried, coheres sufficiently well to be transported. Bamboo stems are cut into pieces 1—5 mm. in thickness and boiled for 10 hrs. at 95—100° with about 35% of lime on the weight of dry bamboo. The product is washed, ground with water for 1·5—2 hrs., pressed into cakes containing approximately 50% of water, and dried. This crude pulp may be subsequently converted into paper pulp by digestion with 6—9% of caustic soda (calculated on dry material) for about 4 hrs. at a pressure of 2 atm. followed by treatment with 25—50% of bleaching powder. D. J. NORMAN.

Treatment of wood and like porous material. M. SMITH and P. PHIPPS (E.P. 265,643, 17.8.25).—Woods and like porous material, particularly those varieties of wood which are not naturally suitable for the manufacture of articles such as lead pencils, are seasoned, softened, and uniformly tinted throughout by impregnating, *in vacuo* if desired, the material, previously dried at 105—127°, with a softening medium, *e.g.*, an 18% emulsion of Turkey red oil, and subjecting it to a comparatively high temperature so that the softening agent volatilises and completely penetrates the interstices of the wood. Suitable apparatus is described. D. J. NORMAN.

Production of moulded pieces from dry mixtures of cellulose derivatives and albuminous substances. F. SCHMIDT (E.P. 248,781, 8.3.26. Conv., 6.3.25).—Albuminous substances are kneaded with cellulose derivatives in the presence of suitable swelling agents, *e.g.*, water in admixture with volatile or non-volatile solvents. The resulting homogeneous mixture is dried, powdered, and subjected to heat and pressure in moulds of the desired shape. *Example*: An intimate mixture of glue (30 pts.), water, and ethylene chlorohydrin is incorporated with a homogeneous cellulose acetate composition containing 50 pts. of cellulose acetate, 20 pts. of water, and 20 pts. of methyl phthalate.

Fillers and hardening agents, *e.g.*, formaldehyde, may be added. D. J. Norman.

Manufacture of felt. C. and E. Pichard (U.S.P. 1,622,883, 29.3.27. Appl., 12.2.26. Conv., 28.2.25).—See E.P. 248,343; B., 1926, 532.

Effecting agglomeration in paper pulp and other suspensions and mixtures. K. Sveen (U.S.P. 1,622,474, 29.3.27. Appl., 17.7.25. Conv., 21.7.24).—See E.P. 237,292; B., 1925, 843.

Pumps for artificial silk spinning machines. P. Hillebrand and A. Olson (E.P. 267,265, 25.1.26).

Artificial silk spinning or like machines. J. L. Rushton and H. Hill (E.P. 266,821, 5.12.25).

Artificial silk spinning machines. J. L. Rushton and J. Lever (E.P. 266,835, 8.12.25).

Apparatus for drying fibrous materials such as fabric, leather, yarns and the like. Barrow, Hepburn, & Gale, Ltd., and A. Hawkyard (E.P. 266,464, 27.11.25).

Softening, spinning, and twisting artificial silk. R. C. Boger (E.P. 266,438, 23.11.25).

Paper-making machines. India Rubber, Gutta Percha & Telegraph Works Co., Ltd., and B. Walkley (E.P. 266,465, 27.11.25).

Machine for drying or chemically treating continuous lengths of fabric or paper. A. Lambrette (E.P. 266,649, 13.12.26. Addn. to 255,297; B., 1926, 740).

Drying cylinders (E.P. 266,481).—See I.

Purification of caustic soda solutions from viscose treatment (E.P. 265,126).—See VII.

Alloys (G.P. 435,170).—See X.

Solvent for cellulose ester (E.P. 252,203).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Patents.

Producing fast coloured resists under Aniline Black. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, und Brüning (E.P. 247,211, 5.2.26. Conv., 5.2.25).—An enol ester ("indigo-sol") of a leuco-vat dye, to which is added ammonium oxalate or other substance producing an acid reaction when steamed, is printed on material padded with aniline. The material is then steamed, passed through a weak dichromate bath, washed, and soaped. Under these conditions the leuco-ester is oxidised in preference to the aniline, and so produces coloured resists under Aniline Black. C. Hollins.

Dyeing leather with acid azo dyes. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, und Brüning (E.P. 247,187, 1.2.26. Conv., 3.2.25).—Full brown shades are obtained on leather by using polyazo dyes of the type: amine (I) → mono- or di-sulphonated naphthylamine or aminonaphthol (II) → non-sulphonated dihydric phenol (III) such as resorcinol ← amine (IV), the amines (I and IV) being any diazotisable arylamines, including aminophenols, aminoazo compounds, and their carboxylic or sulphonic acids. The dyeing of chrome leather with the dye, picramic acid → H-acid → resorcinol ← *p*-nitroaniline, is described. Other examples are *o*-toluidine-5-sulphonic acid or sulphanilic acid or naphthionic acid with H-acid, resorcinol, and *p*-nitroaniline; sulphanilic acid → Cleve's acids → resorcinol ← *p*-nitro-*o*-aminophenol; *p*-nitro-*o*-aminophenol → Cleve's acids → resorcinol ← naphthionic acid; and picramic acid → H-acid → resorcinol ← picramic acid. C. Hollins.

Machine for washing, dyeing, or treating material. E. C. R. Marks. From Max Ams Chemical Engineering Corp. (E.P. 266,551, 25.3.26).

[Vat for] dyeing knitted, woven, or the like articles. C. Callebaut and J. de Blicquy (E.P. 266,227, 30.11.25).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Determination of water content in concentrated sulphuric acid by thermometric titration. T. Somiya (J. Soc. Chem. Ind. Japan, 1927, **30**, 106—112).—When fuming sulphuric acid is added to concentrated sulphuric acid containing a small percentage of water, the temperature of the solution rises, but when the water content is exhausted no further heat is evolved by the continued addition of the fuming acid. By tracing curves correlating temperature elevation and amount of fuming acid, two curves are obtained and their point of intersection gives the end point of the reaction, by which the amount of water in the concentrated acid is determined. Fuming acid is gradually added from a burette into concentrated acid contained in a test tube inserted into a Dumas flask, and the temperature elevation of the mixture is measured. The method is best applied when the concentration of the sample is 90% or more. The error is 0·2—0·5%, according to the amount of the material taken. Commercial fuming acid of any concentration can be used as the standard solution, but that of 10—25% is most suitable, due to its low m.p. The standardisation should be carried out thermometrically with 80% sulphuric acid. K. Kashima.

Sulphur from gas. Cundall.—See II.

Magnesium carbonate for rubber. Tanaka.—See XIV.

Patents.

Concentration of nitric acid. M. Kaltenbach (Addn. No. 30,931, 11.9.25, to F.P. 594,865; B., 1926, 236).—The sulphuric acid used in the prior process is completely denitrated before entry into the third element of the concentrator, by passing a small quantity of steam through it in the second element. L. A. Coles.

Apparatus for the purification of impure solutions of caustic soda or the like on osmotic principles. L. Cerini (E.P. 265,126, 16.6.26. Conv., 29.1.26).—Caustic soda containing colloidal impurities from viscose treatment etc. is purified in an iron reservoir containing vertical partitions dividing it into intercommunicating parallel compartments through which the caustic soda circulates. In each compartment are

disposed semi-permeable diaphragms fastened at their edges to form a tubular bag or sack and in communication with each other to form a conduit for water, which may traverse the apparatus in counter-current to the impure solution. The diaphragms are of cotton, linen, or hemp either in the natural condition or parchmentised by acids, alkalis, or salts. W. G. CAREY.

Process and apparatus for drying salt. P. H. MÜLLER (G.P. 435,837, 1.9.23).—Salt is dried in a current of superheated steam in a chamber shut off from the air, in which the salt drops over a series of hot tubes rounded on the lower side and of acute-angled cross-section on the upper side. L. A. COLES.

Heat economy in the production of salt. METALLBANK AND METALLURGISCHE GES. A.-G., and W. GENSECKE (G.P. 435,589, 15.3.24).—The waste heat of the steam generated in a closed pan for evaporating brine is used for heating the next pan of the series, and the steam generated in this pan is mixed with air which has preferably been preheated. L. A. COLES.

Electrolysis of alkali chlorides. F. GERLACH (Addn. No. 30,954, 22.9.25. Conv. 22.12.24, to F.P. 596,918; B., 1926, 284).—Mercury or other fluid metal is transported in the presence of liquids, *e.g.*, water, alkali lyes, salt solutions, etc., by a device comprising a vertical or inclined screw provided above with a curved plate which participates to some extent in the rotation.
J. S. G. THOMAS.

Production of alkali iodides from absorption charcoal containing iodine. N. V. BOMOMAATSCHAPPIJ ARINA (Dutch P. 15,378, 16.4.25).—The charcoal is treated with approximately the theoretical quantities of an alkali bisulphite solution and an alkali carbonate, and extracted with water. L. A. COLES.

Production of azoimide solution from its alkali salts. DEUTSCHE GASGLÜHLICHT-AUER-GES.M.B.H., Assees. of J. EINIG (G.P. 435,654, 8.9.23).—Alcohol and oxalic acid in sufficient quantity to precipitate sodium dioxalate or potassium dioxalate or tetroxalate are added to a solution containing an alkali azide, and the precipitate is removed. L. A. COLES.

Production of nitrates and a dry mixture of chlorine and nitrosyl chloride. W. DOMINIK (F.P. 611,652, 24.2.26. Conv., 11.3.25).—Metal chlorides, *e.g.*, potassium chloride, are treated with an excess of hot nitric acid containing more than 440 g. HNO_3 per litre, under such conditions that there is a difference of not less than 7 g.-equivalents per litre between the concentration of the nitric acid and that of the chlorine ions. L. A. COLES.

Production of alkali bisulphites or metabisulphites from alkali sulphates and alkaline-earth bisulphites. CHEM. FABR. KALK G.M.B.H., H. OEHME, and E. HERRMUTH (G.P. 435,528, 5.11.25).—Alkali sulphates are added in small quantities to a solution or suspension of an alkaline-earth bisulphite.
L. A. COLES.

Preparation of a mixed salt of calcium phosphate and carbonate for nutritive purposes. C. MÜNSTER and K. THORMANN (G.P. 398,093, 30.11.21).—After the acid phosphate solution has been treated with sufficient milk of lime to transform the free phosphoric acid into monocalcium phosphate, it is thoroughly mixed with the calculated amount of calcium carbonate required to yield a mixed salt with a definite phosphoric acid content. F. R. ENNOS.

Manufacture of active colloids. I. G. FARBENIND. A.-G. (E.P. 255,863, 16.7.26. Conv., 22.7.25).—Active silica is made from a silica jelly containing at least 50 g./litre of silica and no free alkali, by drying the jelly rapidly above 120° with hot combustion gases in a channel or revolving furnace, and heating the mass to incandescence. The process is applicable to all colloids the gels of which can be retransformed into hydrosols. W. G. CAREY.

Drying and grinding Glauber's salt. G. POLYSIUS EISENGIESSEREI U. MASCHINENFABR. (G.P. 435,725, 27.9.23. Addn. to G.P. 398,048; B., 1925, 10).—The salt leaving the drying drum passes direct into an attached grinding mill. L. A. COLES.

Extraction of beryllia from minerals. L. PETIT-DEVAUCELLE (F.P. 611,095, 20.5.25).—The powdered mineral is treated with sufficient alkali hydroxide solution to yield a solution containing an aluminium beryllium alkali trisilicate, and the solution is filtered, treated with carbon dioxide to precipitate silica and alumina, which are removed, and then boiled to precipitate beryllia. L. A. COLES.

Production of beryllium oxide. O. LÉONARD (F.P. 611,457, 9.6.25).—Minerals containing beryllium and hydrofluoric acid, fluorides, or difluorides, are treated successively with sulphuric acid and with excess ammonium carbonate, and the solution obtained is boiled. L. A. COLES.

Treatment and digestion of natural aluminium hydroxides. F. KLIENMANN, and BÜTTNER-WERKE A.-G. (E.P. 266,225, 25.8.25).—Unroasted bauxite is ground with caustic alkali solution in a wet-grinding mill, the resulting fine sludge is treated with chlorine or hypochlorite to free it from organic impurities, and the aluminate–hydrate solution is filtered. The whole operation may be conducted in a ball mill with a long tube so that the alkali and oxidising agents are admitted during the operation, the filtration taking place through the outlet end of the tube, which is pierced and fitted with a filter. The caustic alkali and chlorine may be supplied by an electrolysed solution of sodium chloride.
W. G. CAREY.

Preparation of hydrogen cyanide. I. G. FARBENIND. A.-G. (Swiss P. 115,702, 6.4.25. Conv., 25.8.24).—Mixtures of methyl formate and ammonia are led at 200° over a catalyst, *e.g.*, activated alumina, giving much hydrogen cyanide together with formamide.
C. HOLLINS.

Preparation of sulphur monochloride. E. TERLINCK (F.P. 611,141, 27.5.25).—Chlorine is passed into a hot or boiling solution of sulphur in sulphur dichloride.
J. S. G. THOMAS.

Separation of a mixture of hafnium and zirconium.—W. J. TENNANT. From N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 266,800, 28.11.25).—A mixture of hafnium and zirconium phosphate is introduced into

a medium containing hydrofluoric acid or a difluoride, and is fractionally decomposed by adding a soluble zirconium salt which withdraws the fluorine from the mixture, the hafnium and zirconium phosphates being precipitated, giving at each fractionation a mixture richer in hafnium. W. G. CAREY.

Decomposition of hydrogen sulphide and its removal from industrial gases. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 267,018, 17.9.26).—The hydrogen sulphide is oxidised by bringing pulverised active carbon into suspension in the gases in a disintegrator mill, the gases then being freed from carbon and sulphur by passing them through a direct-current, high-tension field. The carbon charged with sulphur is regenerated by contact with heated surfaces arranged in cascade, or by sprinkling it while upon a travelling band with a solvent for sulphur. W. G. CAREY.

Manufacture of sulphuric acid. J. V. SKOGLUND (E.P. 266,397, 23.9.25).—See U.S.P. 1,559,292; B., 1926, 12.

Production of phosphoric acid. H. A. BRASSERT, W. H. WAGGAMAN, and H. W. EASTERWOOD (U.S.P. 1,622,082, 22.3.27. Appl., 23.1.24).—See E.P. 228,185; B., 1925, 846.

Manufacture of hydrocyanic acid. W. GLUUD (U.S.P. 1,622,372, 29.3.27. Appl., 18.4.24. Conv., 23.4.23).—See G.P. 410,418; B., 1925, 757.

Production of phosphorus pentoxide. G. PISTOR, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,622,206, 22.3.27. Appl., 8.12.25. Conv., 30.12.24).—See G.P. 426,388; B., 1926, 584.

Treating leucite and other sodium and potassium silicates with lime for extracting potassium or sodium aluminates. F. JOURDAN (U.S.P. 1,620,212, 8.3.27. Appl., 27.12.24. Conv., 28.12.23).—See E.P. 226,819; B., 1925, 630.

Apparatus for making anhydrous metallic chlorides. F. T. WOHLERS (E.P. 266,437, 23.11.25).—See U.S.P. 1,564,302; B., 1926, 126.

Process for dissolving a mixture of hafnium and zirconium phosphates and for separating hafnium and zirconium. A. E. VAN ARKEL and J. H. DE BOER, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,618,494, 22.2.27. Appl., 4.1.26. Conv., 6.6.24).—See E.P. 235,217; B., 1925, 713.

Manufacture of cyanides (E.P. 266,809).—See II.

Hydrocyanic acid as an insecticide (G.P. 435,714).—See XVI.

Mercury vapour for inhalation purposes (G.P. 435,689).—See XX.

VIII.—GLASS; CERAMICS.

Surface devitrification of glass. III. Devitrification of alkali silicates. IV. Devitrification of alkali lead silicates. K. TABATA (J. Soc. Chem. Ind. Japan, 1927, **30**, 37—42, 43—47. Cf. *ibid.*, **29**, 473).—The surface devitrification of alkali silicates having the composition of $R_2O,xSiO_2$ is examined. Mixtures of a silicate (97% SiO_2 and 2·75% Al_2O_3) with potassium and sodium carbonates are melted in a furnace. When the temperature of the mixture is above 1000°, the melt is taken out and quickly cooled in the air. Two pieces of glass from the same sample are heated in a muffle furnace at 625—750° for 2 hrs., one of them being lightly scratched with quartz before treatment. After heating, it is quickly cooled and examined under a microscope. According to the mode of crystal production on the glass surface, the devitrification is divided into five classes: (*a*) No crystals; (*b*) a few minute crystals at specified places, such as at the sharpest edges of cicatrixes; (*c*) many crystals along sharp edges, or at the cicatrised portion; (*d*) crystals on all the surfaces having neither sharp edges nor cicatrixes; and (*e*) crystals entirely cover the surfaces. Curves are plotted, showing the relation between the number of molecules of silica dissolved and the percentage K_2O in the alkalis. The curve connecting the points of zero degree is a straight line called the "boundary line of devitrification" or "saturation line of silica dissolution." The compounds present in the melts are assumed to be always metadisilicates. The dissolving power of each mol. of $Na_2O,2SiO_2$ and $K_2O,2SiO_2$ for silica is about 0·5 and 4·5 mols. respectively, at the temperature of the experiment. The dissolving power of mixed alkali silicates for silica is entirely additive. The remarkable differences in the behaviour of both alkali silicates towards silica are considered to be due to the different atomic volumes of the atoms of sodium and potassium. A series of glasses with the composition $R_2O,PbO,xSiO_2$ were tested by the foregoing method, and similar curves have been plotted. Points of the same degree of devitrification give straight lines respectively. The zero degree line runs parallel to the corresponding line for alkali silicates. The composition is suggested for the alkali-lead silicate glasses $x(R_2O,2SiO_2)+y(PbO,2SiO_2)+2SiO_2$. K. KASHIMA.

Importance of silica conversions in the burning process and in the behaviour of refractory materials in industry. E. STEINHOFF (Gas- u. Wasserfach, 1927, **70**, 237—240, 264—268).—The thermal conversions of silica are reviewed and classified into two groups:—(*a*) Non-reversible conversions occurring after prolonged heating or by the aid of catalysts and accompanied by considerable volume change and alterations in crystal structure, *e.g.*, quartz$\rightarrow$cristobalite, 1300—1350°, expansion 17·4%. (*b*) Reversible conversions occurring practically instantaneously, but with little volume change or alteration in crystal structure, *e.g.*, α-quartz $\rightleftharpoons\beta$-quartz, 575°, expansion 2·4%. Reference is made to the anomalous behaviour of β-quartz in passing directly into cristobalite, and to the variable conversion temperature of $\alpha\rightarrow\beta$-quartz (220—260°), which is attributed to the presence of non-volatile impurities. The velocity of the different conversions is largely determined by whether the silica is present in the free state or in solid solution, and also varies inversely as the size of the particles. In the manufacture of silica refractories the greater part of the quartz is converted to cristobalite, but complete conversion is only effected by prolonged heating at 1400—1450°. The reversible changes which introduce expansion and contraction mainly take place below 600°, the reversible expansion being greatest with cristobalite and least with tridymite.

The use of a mixture of converted and semi-converted silica frequently results in the formation of cracks and fissures, owing to the expansion of the non-converted material, and these allow of the penetration of slag and dust. Kieselguhr is converted into cristobalite by heating at 1100—1150°, and, after conversion, its resistance to load between 1000° and 1700° is almost equal to that of 95% silica material.

H. D. GREENWOOD.

Terra cotta. H. SPURRIER (J. Amer. Ceram. Soc., 1926, 9, 773—778).—The structure and physical characteristics of different varieties of terra cotta showed wide variations. No agreement appears to exist as to what constitutes the best terra cotta. A microscopical study showed great differences in the number of voids in a given area; their existence was associated with coarse grog particles. The heterogeneity of the grog was frequently the cause of a number of defects. In the preparation of thin sections for the microscope, the tendency of the sections to curl was taken as evidence of internal strain in the body. Osmosis is not an important factor in the breakdown of terra cotta over large areas. On the other hand, freezing and thawing of wet terra cotta caused marked internal deterioration of the body; this was confirmed by porosity tests. F. SALT.

Colouring of stoneware with cobalt sulphate. A. F. ALZNER (Keram. Rundsch., 1926, 34, 650; Chem. Zentr., 1926, II, 2995).—The calculated amount of cobalt must be added to mask the colour of the iron, together with a suitable excess to act as a colour. The iron content of the clay must be uniform.

B. W. CLARKE.

Characteristics of pyrometric cones. C. O. FAIRCHILD and M. F. PETERS (J. Amer. Ceram. Soc., 1926, 9, 701—743).—The characteristics of Orton cones were determined under standardised conditions of heating resembling those occurring in ceramic practice. An electric tube furnace was used for the experiments with cones fusing below 1200°, and a graphite spiral furnace was designed for the more refractory cones. Means were provided for varying the nature of the atmosphere within the furnaces. Thus the experiments were carried out under definite and reproducible conditions. The temperatures of the "end-points" and of the bending intervals of a complete set of cones were measured in clean air and in various kiln gases. With few exceptions, the end-points were higher when the rate of heating was 150°/hr. than when it was 20°/hr. Under slow heating in gases free from sulphur dioxide, the end-points of cones 015—01 were higher than in air; kiln gases free from sulphur dioxide had little effect upon cones 022—016 and 1—42. The accuracy of the method developed in this investigation was within 5° up to 1400° and within 10° from 1400° to 2000°. The end-point of cones is variable, depending upon time, temperature, and atmosphere. Given a constant firing schedule and kiln atmosphere, a cone will always come down at the same temperature. Hence cones measure the temperature at which ware will mature under given rate of heating and character of kiln gases. The combined use of cones and pyrometers is recommended for kiln control, but cones are inherently unsuited for the determination of the refractoriness of clays in laboratory tests. F. SALT.

Comparison of the softening points of some foreign and American pyrometric cones. R. F. GELLER and E. E. PRESSLER (J. Amer. Ceram. Soc., 1926, 9, 744—757).—The results of a comparative study of the softening points of English, French, German, and American cones are presented graphically, and by means of drawings and photographs. The softening points of cones 022—20 were determined in a gas-fired, recuperative, down-draught kiln, whilst a graphite resistance furnace was used for cones 26 to 35. F. SALT.

Silicon carbide refractories for water-gas generators. M. L. HARTMANN and J. A. KING (J. Amer. Ceram. Soc., 1926, 9, 758—765).—The rapid deterioration of ordinary firebrick linings in water-gas generators is due mainly to clinkering trouble. Clinker adhesion and slag erosion have been largely eliminated by the use of Bernitz blocks made of "Carbofrax" (bonded silicon carbide). These are hollow, square, or rectangular shapes pierced with a number of holes which taper inward from the face nearest the fire. Air or steam can be passed through these holes at various points into the fire. The success of these blocks when used for lining water-gas generators is due to their great mechanical strength at high temperatures, lack of chemical activity in contact with viscous slags, and high thermal conductivity. F. SALT.

Harper electric kiln. F. A. J. FITZGERALD (J. Amer. Ceram. Soc., 1926, 9, 766—772).—The construction of an electric tunnel kiln, 68 ft. long, designed for firing biscuit ware, is described. A temperature of 1500° has been attained in the kiln when firing porcelain.

F. SALT.

PATENTS.

Process and apparatus for the production of silica articles. H. L. WATSON, Assr. to GENERAL ELECTRIC Co. (U.S.P. 1,621,446, 15.3.27. Appl., 1.4.26).—Silica is shaped by heating to fusion a part only of the mass, so that the unfused silica serves as a support for the fused silica. The fused silica is then withdrawn, leaving behind the unfused mass. W. G. CAREY.

Production of white opacifying media for glasses and enamels. I. KREIDL (E.P. 245,757, 29.12.25. Conv., 10.1.25).—Uncoloured oxides and compounds of metals other than tin and zirconium are converted into the colloidal gel state in a colloid mill, and are maintained in a highly polymerisable form, even at the annealing temperature of the enamel, by the addition of tin or zirconium compounds as protective substances.

W. G. CAREY.

Manufacture of ceramic materials, building elements, and the like, from mud. A. LOESSIN (E.P. 259,236, 1.10.26. Conv., 1.10.25).—Mud deprived of about one half of its water by settlement for some 4 months and decanting, or by centrifugal action, is kneaded in a kneading machine with Glauber's salt, with or without sand, and after preliminary burning at 400—500° is cooled and given a final burn, forming ceramic products. Clinker is made by giving a two-stage preliminary burning with intermediate cooling and a final burning. Paving is formed by a final burning at

1300°, whilst a thin flowing casting material is produced at 1350—1400°. W. G. Carey.

Quartz working. E. R. Berry, Assr. to General Electric Co. (U.S.P. 1,620,511, 8.3.27. Appl., 18.4.23). —See E.P. 214,630; B., 1924, 713.

Treatment of earthy minerals. W. Feldenheimer (U.S.P. 1,622,099, 22.3.27. Appl., 30.10.25).—See E.P. 242,358; B., 1926, 55.

Methods and apparatus for forming sheet glass. L. Mellersh-Jackson. From Hartford-Empire Co. (E.P. 266,182, 31.5.26).

Manufacture of double-walled glass flasks. F. Siegheim (E.P. 267,005, 1.9.26).

IX.—BUILDING MATERIALS.

Patents.

Burning cement. H. Kühl (Austr. P. 104,148, 31.3.25. Conv., 20.1.25).—An oxygen carrier is added to a finely pulverised mixture of raw materials for cement and fuel, in order to facilitate the combustion of the fuel. Thus, *e.g.*, ferric oxide is added to the raw materials to make the proportion one twelfth that of the lime content. B. W. Clarke.

Burning cement and the like. M. Büttner (G.P. 435,077, 29.8.24).—A rotary kiln is provided with one or more constrictions, which form a sintering zone or "condensing" chamber, into which penetrates the fuel inlet pipe, having a large surface area. The speed of rotation of the kiln is arranged so that the product of the square of the number of rotations per min. and the greatest internal diameter in metres of the lining of the calcining zone is 500 or more. The process results in a considerable lowering of the coal consumption and of the waste-gas temperature. B. W. Clarke.

Rotary tubular kilns for treating cement and the like. O. Bouzin (E.P. 266,939, 4.5.26).—A metal heat exchanger, arranged in the preheating zone of the kiln, is provided with a series of radial plates at the periphery which spread the material to be treated in thin sheets over a central tube or bundle of tubes through which the hot gases pass. The inner wall of the calcining zone has ring-shaped projections which spread the material along a succession of horizontal planes, thereby impeding its progress and increasing the capacity of the kiln. The tubular clinker cooler is provided with a similar heat exchanger, and the amount of air admitted is limited to that strictly necessary to maintain combustion in the kiln. B. W. Clarke.

Apparatus for preheating slurry, especially cement slurry. G. Polysius, Eisengiesserei u. Maschinenfabr. (G.P. 435,792, 24.2.24).—The slurry inlet pipe passes through the smoke chamber, where it is heated by the waste kiln gases, and is connected to the kiln by a pipe provided with a jacket through which the waste gases are conducted. This prevents the heat taken up by the material in the smoke chamber from being lost during its passage to the kiln. B. W. Clarke.

Manufacture of acid-proof cementing compositions. I. G. Farbenind. A.-G., Assees. of Farbwerke vorm. Meister, Lucius, & Brüning (E.P. 256,258 and 258,616, [A], 30.7.26, [B], 18.9.26. Conv., [A], 30.7.25, [B], 18.9.25).—(A) A powdered modification of silicic acid, such as opal, chalcedony, etc., which is capable of reacting strongly with alkali and will show a loss of weight of not less than 40% of its original weight when boiled for 2 hrs. with 25 times its weight of 15% sodium hydroxide solution, is added to a water-glass solution forming an acid-proof cementing composition. Powdered fireclay or other similar fillers may be added to increase the binding power. (B) The addition of 3—4% of powdered silicon to the above or other water-glass cement produces an acid-proof composition with a thermal conductivity comparable with that of metals, thereby increasing the durability of the cement when used with metal vessels. B. W. Clarke.

Production of cementitious material. A. E. Hills (E.P. 266,775, 20.10.25).—A suitable proportion of the hydrated oxide of iron or aluminium is added to hydraulic cements, especially Portland cement, to combine with the lime set free on setting, thereby accelerating the maturing of the cement and preventing efflorescence. The mixture is suitable for use in manufacturing cement-asbestos sheets, tiles, etc. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 24,718/1908 (cf. B., 1909, 984).] B. W. Clarke.

Insulating and building materials. Soc. Anon. Cimenti (Swiss P. 115,884, 19.3.25).—Vegetable fibres, *e.g.*, straw, maize, wood fibre, etc., rendered fireproof with solutions of magnesium salts or water-glass, are mixed with materials, such as Portland cement or plaster, which will react with the moisture in the fibrous material, the mixture is poured into moulds, and removed to complete the hardening process after a sufficient preliminary setting. B. W. Clarke.

Porous compounds or compositions formed from gypsum. G. O. Case (E.P. 266,524, 3.2.26).—Calcium carbonate is added to partly dehydrated gypsum, or natural gypsum rock containing 3—13% of calcium carbonate is calcined until about 6·5% of water is left, and either product is treated with hydrochloric or other acid of *d* 1·02 to the extent of 40—60% by wt. of the dehydrated gypsum. The mixture is cast in moulds having porous tops. The resulting blocks may be made waterproof by a layer of cement or concrete, or by the addition of ferric or magnesium sulphate to the mixture. W. G. Carey.

Manufacture of priming material for coating porous surfaces. E. W. Frenkel and A. J. H. Brust (E.P. 266,401, 26.9.25).—A stable suspension of finely-divided light magnesia, magnesium carbonate, levigated alumina, etc. in linseed oil, the suspensoid condition of which is maintained by adding an altered fatty oil such as coagulated wood oil or oxidised linseed oil, is used for priming absorbent material. The operation may be performed by altering linseed oil while in admixture with the pore filler in a mill by oxidation or by sulphur chloride, and stopping the reaction when desired by adding more linseed oil together with turpentine substitute as thinning agent and siccatives if desired. W. G. Carey.

Manufacture of tarred macadam and the like. J. F. Wake (E.P. 266,420, 30.10.25).—The agglomeration in cold weather of tarred macadam aggregate is prevented by the addition of a coating of petroleum, linseed oil, or other film-producing substances, the lubricant being introduced by spraying or sprinkling the aggregate. W. G. Carey.

Kiln for burning cement. C. Naske (U.S.P. 1,622,337, 29.3.27. Appl., 7.2.25. Conv., 8.1.24).—See E.P. 227,444; B., 1926, 409.

Production of artificial stones. J. Jakob (E.P. 244,724 and 266,789. Appl., [A], 3.11.25, [B], 23.11.25. Conv., [A], 22.12.24).—See G.P. 417,360; B., 1926, 130.

Compositions for laying or preventing dust and their application to road surfaces. P. G. Ekström (E.P. 252,378, 19.5.26. Conv., 22.5.25).—See U.S.P. 1,606,928; B., 1927, 110.

Manufacture of cement concrete and apparatus for use therewith. A. C. Knipe (E.P. 266,814, 3.12.25).

Building materials from mud (E.P. 259,236).—See VIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Chromium-cobalt steels. F. M. Ostroga (Rev. Mét., 1927, **24**, 135—145; cf. B., 1927, 77).—Two steels containing 1·21% C, 13·16% Cr, 4·88% Co, 0·64% Mo, 0·59% Ni and 1·78% C, 13·06% Cr, 2·40% Co, 1·14% Mo, 0·11% Ni, used for exhaust valves for internal-combustion engines have been studied by dilatometric and microscopical methods and their Brinell hardnesses determined. The steels are not readily oxidisable at high temperatures, the transformation temperature on heating is 850°, and the hardness is 60 at 800°. In the malleable condition they are easily machineable. That the carbon content is less towards the edge than at the centre of the rod is regarded as a favourable factor in the application of the steels to exhaust valves, and in spite of their fragility, which may be accentuated by the presence of impurities or eutectic formed by heating above 1200°, chromium-cobalt steels have under certain conditions given satisfactory results. M. Cook.

Silicon construction steel from the Siemens-Martin furnace. J. Meiser (Stahl u. Eisen, 1927, **47**, 446—448).—Particulars are given of eight heats of silicon steel made in an 80-ton tipping open-hearth furnace at the Dortmund Union Works for bridge construction. The analyses had the following limits:—0·13—0·19% C, 0·80—1·37% Si, 0·84—1·14% Mn. Also at a Rhenish works, 6 heats were made in a smaller (17-ton) furnace of a steel for railway wagon construction, with analysis range: 0·15—0·19% C, 0·85—1·10% Si, 0·90—1·10% Mn. The results of a large number of acceptance tests show that such constructional steels can be satisfactorily produced in ordinary open-hearth furnaces. T. H. Burnham.

Advantages of smelting fine-grained ores in the blast furnace. K. Hofmann (Trans. Amer. Electrochem. Soc., 1927, **51**, 91—102).—In the reduction of ferric and ferrosoferric oxides by hydrogen, the degree of reduction is a minimum at 750° owing to surface welding of the particles already reduced; this action commences at about 600° and prevents the diffusion of the hydrogen into the interior. At temperatures below 600° ferric oxide is much more readily reduced than ferrosoferric oxide of the same grain size, an indication that the latter is not an intermediate product of the reduction of ferric oxide to metallic iron. In the case of iron ores, similar results are obtained, but the retardation of the action above 600° is intensified by the presence of silica, which forms ferrous silicate at 700° with the ferrous oxide produced in the reduction, and this forms a glassy, impervious coating on the oxide particles. If fine iron ores, therefore, could be used in the blast furnace in such a way that they were completely reduced at a relatively low temperature, the height of the furnace could be reduced and the boshes and hearth could be cylindrically shaped and of the same diameter. A. R. Powell.

Gaseous cementations. Cementation by cyanogen. II. M. Pérot (Rev. Mét., 1927, **24**, 79—92; cf. B., 1927, 45).—Examination of test pieces of a steel containing 0·2% C, which have been heated for varying periods in cyanogen at 1000°, shows that rapid cementation takes place during an initial time of 45 min., and that the rate then quickly diminishes. A layer of non-assimilable carbon is deposited on the specimens, and carbon slowly diffuses towards the interior from the hypereutectoidal zone already rich in carbon. Evidence is obtained to show that renewal of the cyanogen atmosphere increases the rate of cementation. Cementation is little affected by variations in cyanogen pressure from 767 to 60 mm. of mercury. Values are given for the depth of cementation by cyanamide with varying times and temperatures of heating. Sodium cyanide is not useful as a cementation agent, since its fusion at a comparatively low temperature appears to stop the reaction. L. M. Clark.

Existence of limiting states in alloy studies. Equilibrium diagram involving the existence of limiting states. L. Grenet (Rev. Mét., 1927, **24**, 93—107).—A theoretical investigation of limiting states in alloys. The anomalous behaviour of alloys (such as is observed in the iron–nickel series) is examined from the point of view of the existence of critical points in phase equilibria and transformations. L. M. Clark.

Recrystallisation [of iron, steel, and copper]. H. Hanemann (Stahl u. Eisen, 1927, **47**, 481—489).—The rate of recrystallisation of iron, which has been deformed by hammering at a high temperature and immediately annealed at the same temperature, is a logarithmic function of that temperature, the equation to the recrystallisation curve being $R = 1{\cdot}016^{(T-500)}$, when the degree of deformation is constant. During recrystallisation, the average number of new grains per unit area decreases hyperbolically with the time of annealing, and is smaller, after any given time, the higher the temperature of annealing. As is the case with copper, there is a certain minimum degree of deformation to which iron and steel must be subjected before recrystallisation will ensue on annealing; this minimum value decreases hyperbolically with rise of temperature up to the transformation point. There is also a minimum

temperature of annealing below which no recrystallisation will take place, however severe the deformation; in the case of soft iron this is 450° in the α range and 910° in the γ range. Parameter curves for the recrystallisation of copper, iron, and steel together with the corresponding equations are given, also curves showing the grain size of iron after cold deformation and recrystallisation, and of soft iron forged at temperatures between 900° and 1100°. A. R. Powell.

Working of steel containing copper. W. Herwig (Stahl u. Eisen, 1927, **47**, 491—493).—The presence of copper in steel sheets renders them unsatisfactory for use in the manufacture of deeply recessed articles, but such steel may be used in making heavy parts of machinery without fear of fracture. Steel sheets containing much copper acquire a rough, hackly surface during rolling, and it is difficult to remove the scale from their surfaces. Cupriferous steel is much more easily corroded than steel free from copper, and, in the case of open-hearth steel, the rate of corrosion is roughly proportional to the copper content. A. R. Powell.

Influence of the [heat and mechanical] treatment of transformer iron on the wattage losses. G. Eichenberg and W. Oertel (Stahl u. Eisen, 1927, **47**, 262—269).—The effect of various methods of rolling and annealing transformer iron with 4% Si on the wattage losses has confirmed the observations of Yensen (Bull. Univ. Illinois, No. 83, 1915) that the losses are smaller with sheets consisting of large, evenly distributed crystallites than with those containing unevenly oriented and irregular crystal grains. The effect of grain size, however, is often masked by other influences such as the so-called "gas content." Prolonged annealing results in the interaction of oxide inclusions with the carbon content, and the subsequent elimination of the carbon monoxide so formed results in an appreciable decrease in the wattage loss. This beneficial action is obtained by annealing at 750° for the final rolling operation.

A. R. Powell.

Sulphur in iron and steel. J. Ciochina (Chim. et Ind., 1927, **17**, 383—384; cf. B., 1927, 167).—Free sulphur in iron and steel is determined by passing carbon dioxide through a Corleiss flask, in which the sample (10—20 g.) is being dissolved in concentrated hydrochloric acid, for 2 hrs., during which time the flask is gently heated. The reaction having ceased, the current of gas is continued for ½ hr. to remove any remaining hydrogen sulphide. 25 c.c. of hydrofluoric acid are added and carbon dioxide is again passed for ½ hr. After cooling, excess of potassium hydroxide solution is added, the whole boiled, then cooled, and 200—300 c.c. of concentrated hydrochloric acid are added, the mixture being again heated. Before the acid addition, carbon dioxide is passed into the flask, and, after heating for 1 hr., the contents are titrated with iodine and thiosulphate solution. Results obtained by this method have shown that in iron containing 0·15—0·2% S, 0·005—009% exists as free sulphur, and in slag containing 1·84% S, 0·012% is present as free sulphur. If a sample of iron is ground with mercury before analysis, mercury sulphide is formed, and only traces of free sulphur can be found. The determination of sulphur by combustion gives higher results than the hydrogen sulphide method, for with the latter only the result for sulphur occurring as sulphide is obtained, whereas the former yields the total sulphur. A sample of iron rapidly cooled and another sample, from the same ladle, slowly cooled were found to contain 0·148 and 0·106% S respectively. The difference between the two is regarded as sulphur in the gaseous form which, when the iron is quickly cooled, does not escape but combines to form iron sulphide and 3—4% of it remains as free sulphur.

M. Cook.

Determination of small amounts of titanium in alloy steels. K. Roesch and W. Werz (Chem.-Ztg., 1927, **51**, 149—150).—The sample (5—10 g.) of turnings is dissolved in 1 : 1 hydrochloric acid. If tungsten is a constituent of the steel the tungstic acid which is precipitated contains part of the titanium, and is fused with sodium carbonate. On treating with hot water the sodium titanate is not dissolved, but is treated with hydrochloric acid which may be added to the original solution. Iron is separated by the Rothe ether process. Chromium if present is brought into solution as chromate by treatment with sodium peroxide or carbonate, vanadium with sodium hydroxide, and molybdenum by either of these means. Nickel and cobalt, on treatment with excess of ammonia, pass into solution as hydroxides. The titanium in hydrochloric acid solution is reduced by zinc to the trichloride with exclusion of air, a freshly prepared 50% solution of potassium or ammonium thiocyanate is added, and titrated with 0·3N-ferric chloride solution until a red-brown colour persists. The experimental error of the process is as follows:—Ti = 0·01 — 0·10%, ± 0·003; Ti = 0·10 — 1·00%, ± 0·005. T. H. Burnham.

Crystallite orientation of copper in relation to the degree of rolling. G. Tammann and H. H. Meyer (Z. Metallk., 1927, **19**, 82—84).—On rolling copper the crystallites slip along an octahedral plane and, until a reduction of 30—40% in thickness has been effected, the proportion of octahedral planes in the plane of rolling increases. Further work results in the breaking up of the elongated crystals in the direction perpendicular to the direction of rolling, and finally these lamellæ split up into long, narrow fibres, which are apparently twinned. On etching, these fibres develop a series of parallel lines arranged at an angle of 35° ± 5° to the direction of rolling, the lines running towards that direction and away from it in alternate fibres. Up to about 40% reduction the proportion of octahedral planes on the surface increases, but with 50% reduction they have practically disappeared, giving place to dodecahedral planes. After annealing at 400—500°, the surface crystallites are practically all so oriented that an octahedral plane lies with its apex in the direction of rolling. Severely cold-rolled sheets, however, pass through a transition state in which there is a large proportion of unevenly oriented dodecahedral planes in the surface.

A. R. Powell.

Hardness and potential of zinc–copper alloys. O. Bauer and O. Vollenbruck (Z. Metallk., 1927, **19**, 86—89).—The hardness of zinc–copper alloys containing up to 35% Zn is very little greater than that of pure copper. Between 35% and 39% Zn the hardness

rises to almost double its previous value, then remains stationary again throughout the $\alpha + \beta'$ range. In the range of pure β' (47—49%) an abrupt increase in hardness takes place, and this continues to a maximum of 310 at 61% Zn, corresponding with the formation of Cu_2Zn_3. After this, further addition of zinc results in a rapid fall in hardness to a third constant value in the $\epsilon + \eta$ range somewhat less than in the $\alpha + \beta$ range; finally, there is a small but abrupt fall to the hardness of pure zinc (48). In a 1% sodium chloride solution at 18° the potential of pure copper measured against the normal calomel electrode is 0·19 volt. Almost the same potential is shown by copper–zinc alloys containing up to 39% Zn, but, as soon as the β' constituent appears, the potential rises to 0·31 volt; then increases steadily to 0·40 volt with pure β' (47% Zn), and falls again to 0·36 volt with 50% Zn, corresponding with the appearance of γ. Within the $\beta + \gamma$, and the γ ranges, a slight but steady increase of potential occurs with rise in the zinc content, followed by a sudden rapid increase to about 1 volt with the appearance of the $\gamma + \epsilon$ constituent (70% Zn). The value then oscillates about this figure for alloys containing 70—100% Zn.

A. R. POWELL.

Gases contained in brasses, aluminium, and its alloys. L. GUILLET and A. ROUX (Compt. rend., 1927, **184**, 724—727; cf. B., 1927, 15).—Gases are evolved from brasses containing copper (60—67%) and zinc, between 530° and 1040°, the maximum rates occurring at 720—790°, and principally at 930—980°. The volume of the gas was half that of the metal, and had the composition CO_2 19, CO 9·5, H 35·9, CH_4 22·2, and N 13·4%. Aluminium containing silicon and iron (0·6%) evolved 0·14 of its volume of gas in 3 hrs. between 400° and 550°, and this had the composition CO_2 20, CO 12, H 68·0%. The physical properties of the metal were the same whether annealing was carried out in air or *in vacuo*. When the metal was heated to 820° at the rate of 7° per minute, the volume of gas was 1·6 times that of the metal, and its composition CO_2 31·7, CO 2·45, H 26·0, CH_4 26·0, and N 3·25%. Duralumin yielded in 2½ hrs. at 500°, 0·23 of its volume of a gas containing CO 9, H 91%.

J. GRANT.

Aluminium as constructional material in the inorganic chemical industry. BUSCHLINGER (Z. Metallk., 1927, **19**, 101—106; cf. B., 1927, 143).—The action of a large number of inorganic compounds on metallic aluminium at various temperature has been examined. Solutions of most salts of the mineral acids, with the exception of alkali nitrates, have a more or less corrosive action on the metal, halides being the most destructive. Dilute ammonia solutions and alkali carbonates have very little action either hot or cold. Sulphur may be melted without danger in aluminium pans, which are also suitable for use with hot ammonium sulphide liquors.

A. R. POWELL.

Structure and tensile properties of very pure aluminium. VON GÖLER and G. SACHS (Z. Metallk., 1927, **19**, 90—93).—The course of recrystallisation of very pure aluminium (99·9% Al) compared with that of technical grades has been examined by means of *X*-ray interference figures. As is the case with technical grades, pure aluminium commences to recrystallise just above 200°, but the crystals grow at a much more rapid rate, so that at 375—400° the structure of the pure metal consists of very large crystals, whereas a similar structure is produced in the technical grades only after prolonged heating above 600°. The curves showing the variation of tensile strength, elongation, and reduction of area, with the annealing temperature run practically parallel for the 99·9, 99·0, and 98·7% grades of aluminium, minimum tensile strength and maximum ductility being obtained in every case at 300°. These results show that even the small quantities of impurities in the 99·9% grade are not completely in solid solution at the ordinary temperature.

A. R. POWELL.

Leaching of molybdenite ores. C. SVENSSON (Trans. Amer. Electrochem. Soc., 1927, **51**, 51—58).—Norwegian molybdenite ore (1·9% MoS_2) occurs as very fine flakes dispersed throughout a silicious dolomitic rock, and is not amenable to concentration by any of the usual methods. Roasting in a revolving tube furnace at 750—800° effected complete conversion of the molybdenum to trioxide, and of the sulphur to calcium sulphate, and subsequent leaching with sodium carbonate solution at 80—90° extracted over 90% of the molybdenum as sodium molybdate, together with most of the sulphate. From the leach liquor, after acidification with sulphuric acid, the molybdenum could be precipitated as sulphide, or, alternatively, it could be recovered as calcium molybdate after removal of the sulphate from the acidified solution with barium chloride and subsequent neutralisation with milk of lime and ammonia. Cost data based on the laboratory results are given.

A. R. POWELL.

Metal calorimeter for determination of the specific heats of metals, oxides, and slags. W. GROSSE and W. DINKLER (Stahl u. Eisen, 1927, **47**, 448—454).—An apparatus is described based on the Nernst metal calorimeter suitable for calorimetric measurements over a temperature range of 0 to 1600°. The specimen is pear-shaped, and is heated in a tubular furnace wound with nickel–chromium wire for use up to 1250°, and above this temperature with molybdenum wire or a carbon spiral *in vacuo*. It falls into a copper vessel the temperature of which is indicated by 20 copper–constantan thermo-couples arranged in series. For molten metals a quartz container is used or a drop is allowed to fall from a conical rod. Calibration is carried out under the same conditions as the sp. heat determinations, water in a thin brass vessel being used as calibrating medium. It is heated by steam, which is also used for determining heat contents up to 100°. The weight of the specimen is such that the same amount of heat is always introduced into the calorimeter. After each experiment the small unavoidable losses of heat are determined. By use of a Kurnakov apparatus the heat transmission from the specimen to the calorimeter is made self-registering.

T. H. BURNHAM.

Damping properties of some metals [iron, copper, and aluminium] in torsional vibration. O. FEUSSNER and E. RAMB (Z. Metallk., 1927, **19**, 115—116).

PATENTS.

Sponge iron. COPPER SEPARATION, LTD., Assees. of P. W. NEVILL (Austral. P. 20,811, 1.12.24).—A mixture of finely-divided iron ore and coal is heated at

900—1000° in a shaft furnace to which a finely-divided air supply is admitted over a large surface so that at the most only a partial fusion of the mass takes place. The hot reduced product is quenched in water, the unchanged carbon washed away, and the iron recovered from the residue by crushing followed by magnetic separation. A. R. Powell.

Tool steel without brittleness. Röchling'sche Eisen- u. Stahlwerke G.m.b.H., and J. Kubasta (Austr. P. 104,009, 18.11.21).—The formation of a metasilicate in the metal is avoided by removing the slag from its surface and allowing the silicon present to separate and the metal to take up oxygen. Subsequently the excesss of ferrous oxide is removed by deoxidation in the usual manner. A. R. Powell.

Heat treatment of steel. E. F. Kenney (U.S.P. 1,619,025, 1.3.27. Appl., 23.4.26).—Steel rails or the like are cooled from a temperature (about 950°) above the critical range, to a temperature (380°) below that range, but above blue-heat, heated at an intermediate temperature (540°) for 1 hr., and allowed to cool in air. The physical properties are greatly improved. T. S. Wheeler.

[Alloy] steel. C. K. Everitt, and E. Allen & Co. (E.P. 267,024, 28.9.26).—An alloy steel of high resistance to corrosion contains less than 2% and, preferably, not more than 1·5% Cu, 7—20% Cr, 4—20% Ni (preferably 6%), 0·05—0·5% C, refractory metals of the carbon group being absent. C. A. King.

Alloy steel. W. H. Keen (U.S.P. 1,621,886, 22.3.27. Appl., 10.5.24).—An air-hardening steel capable of being forged contains, approximately, 3% V, at least 0·85% C, 18% W, and 4% Cr. C. A. King.

Titanium alloy. A. W. Clement, Assr. to Ludlum Steel Co. (U.S.P. 1,621,523, 22.3.27. Appl., 18.12.17). —An alloy containing 10—30% Cr, 5—12% Ti, up to 1% Si, up to 10% Al, together with carbon sufficiently low for working purposes, the remainder being iron. C. A. King.

Manufacture of alloy steel and iron. B. D. Saklatwalla (U.S.P. 1,619,462, 1.3.27. Appl., 16.7.24). —A bath of molten steel or iron covered with slag is treated with successive small portions of chrome ore, or other unreduced compound of an alloying metal, and a reducing agent, *e.g.*, silica in the form of ferro-silicon. T. S. Wheeler.

Composition of matter. [Iron alloy.] R. P. Drummond, Assr. to Pacific Cast Iron Pipe & Foundry Co. (U.S.P. 1,620,877, 15.3.27. Appl., 7.8.25). —The alloy consists of iron in combination with 1·75—2% C, 6·25—8% Mn, and 1·50—2% Si. F. G. Crosse.

Production of iron and other carbon binding metals and alloys thereof with a certain percentage of carbon directly out of oxide ore. H. G. Flodin and E. G. T. Gustafsson (E.P. 243,353, 18.11.25. Conv., 19.11.24).—A charge of a mixture of ore and carbonaceous material containing only sufficient carbon for the reduction process is, during or after reduction, mixed with carbon alone or with a high-carbon ore mixture to increase the carbon content of the metal to the desired amount. When the reduction is effected in an electric furnace, the high-carbon mixture, which may be in the form of briquettes, is introduced on to the surface of the slag around the electrodes. C. A. King.

Chemically resistant alloys [of iron, nickel, and copper]. R. Walter (G.P. 435,170, 13.5.24).—Alloys suitable for use with sulphite-cellulose liquors comprise varying proportions of iron, copper, and nickel with 10% Si. A. R. Powell.

Direct reduction process for producing carbon-binding metal or metal alloy. H. G. Flodin and E. G. T. Gustafsson (E.P. 243,743, 26.11.25. Conv., 27.11.24).—A mixture of oxide ore and carbon together with necessary fluxes is reduced in an electric furnace having electrodes which can be raised or lowered, the heating being effected chiefly near the surface of the slag bath. The charge may be introduced into the furnace continuously, being interrupted for a sufficient time before tapping to allow the metal to be raised to a suitable temperature, during which period the electrodes are immersed in the slag bath. C. A. King.

Conversion of sulphide ores into sulphates. Complex Ores Recoveries Co. (G.P. 434,948, 2.2.22).—Pyritic ores containing copper, lead, or zinc are roasted at 600—1000° to convert the sulphides into oxides, sulphates, and ferrite. The roasted material is then raked in the direction of flow of the sulphurous gases into a cooler part of the furnace so as to favour the formation of copper, zinc, and lead sulphates and to convert the iron into a basic ferric sulphate. A. R. Powell.

Treating sulphide ores of lead and zinc. N. C. Christensen (U.S.P. 1,620,873, 15.3.27. Appl., 26.12.23). —Ores containing galena are agitated with cold acid brine until the liberation of hydrogen sulphide is completed, and lead is then recovered from the solution. C. A. King.

Obtaining blende free from chlorides by melting lead-zinc ores with a mixture of potassium and zinc chlorides. E. Langguth (G.P. 435,704, 16.5.25). —Lead-zinc sulphide ores are melted with a mixture of potassium and zinc chlorides to convert the lead into chloride, which is then reduced to metal by the addition of zinc. The slag is extracted with the minimum of water, and hydrochloric acid is added in quantity just sufficient to dissolve the precipitated zinc oxychloride. The residue after filtration consists of relatively pure blende. A. R. Powell.

Process and apparatus for the condensation of zinc vapours. C. J. G. Aarts (E.P. 249,123, 10.3.26. Conv., 10.3.25).—Zinc vapours are condensed in a vertical duct provided with a number of water-cooled coils which may be regulated individually so as to produce a given temperature in any particular section of the duct. In practice, a number of such ducts may be used in parallel, forming at their lower parts a chamber for collecting liquid zinc, and being connected at the upper ends with a dust collector which discharges the collected dust to a charging device at the base of the condensing ducts. C. A. King.

Shaft furnace for roasting ores and calcining fine-grained materials. G. A. Strecker (G.P. 435,703, 22.2.24).—The flues for the air supply of a

shaft furnace for roasting ores and the like are so arranged that the air passing through the cooling shaft, and there preheated, passes directly into the roasting zone and thence into the hottest zone. Regulating dampers are provided at intervals in the air flues to allow of the admission of fresh air to any desired zone of the furnace. A. R. Powell.

Smelting ores in the absence of air. A. Roitzheim and W. Remy (G.P. 435,109, 26.6.25).—To avoid slagging of the walls of the reduction chamber the ore is mixed with an aqueous suspension of finely-divided carbon or aliphatic hydrocarbon and an inorganic colloid, *e.g.*, clay, and the mixture is charged into the furnace wet or after drying, briquetting, and carbonising. A. R. Powell.

Cadmium plating. Udylite Process Co., Assees. of A. W. Young and M. E. Louth (E.P. 266,985, 28.7.26. Conv., 31.3.26).—In a process of cadmium plating from a cyanide bath, a mixture of an extract of a cereal, *e.g.*, bran, wheat, maize, etc. with sugar is used as an additive agent. An impure sugar, *e.g.*, molasses or the caramelised form, is more suitable than pure cane sugar. C. A. King.

Lead alloys. H. Yoshikawa (E.P. 253,920, 17.6.26).—Binary alloys of lead and bismuth forming a one-phase solid solution contain 4%, 0·25%, and 0·25—0·025% Bi, respectively. The alloys are harder, and have a small coefficient of friction as compared with lead, and are also more resistant to chemical and electro-chemical attack than other lead alloys. C. A. King.

Extraction of gold. J. L. Warner (U.S.P. 1,621,697, 22.3.27. Appl., 19.12.23).—Gold-bearing material consisting of clay and sand is screened dry, the finer portion being crushed and agitated in water to separate the sand from the clay. Washings from the coarser material are added to the sludge from the smaller grained material, and sand is removed from the clay particles. C. A. King.

Making bi-metallic or compound metallic strips, plates, or the like, such as for use in thermostats. F. W. Miller (E.P. 266,645, 26.11.26).—A metal plate is covered with a non-metallic fusible material, *e.g.*, powdered glass, and heated until the glass melts. A plate of metal of lower fusibility than the first is then laid on the glass and melted, causing the glass layer to rise to the top and the two metal layers to become united. C. A. King.

Manufacture of iron–chromium alloys. W. B. D. Penniman and E. J. Shackelford (E.P. 244,413, 3.9.25. Conv., 11.12.24).—See U.S.P. 1,527,088; B., 1926, 363.

Magnetic alloy. W. S. Smith, N. Poppleford, and H. J. Garnett (U.S.P. 1,622,008, 22.3.27. Appl., 2.5.25).—See E.P. 224,972; B., 1925, 76.

Increasing the production and phosphoric acid content of Thomas slag. H. Hilbert (E.P. 247,946, 11.2.26. Conv., 19.2.25).—See G.P. 418,102; B., 1926, 162.

Electrolytic separation of metallic chromium. F. W. Würker (E.P. 266,045, 16.11.25).—See F.P. 607,829; B., 1927, 194.

Solder for aluminium and its alloys. E. Conti (U.S.P. 1,619,852, 8.3.27. Appl., 12.9.24. Conv., 21.7.24).—See E.P. 237,224; B., 1925, 811.

Alloy of lithium and aluminium. J. Czochralski and G. Welter, Assrs. to Allied Process Corp. (U.S.P. 1,620,081, 8.3.27. Appl., 28.6.20. Conv., 15.2.19).—See E.P. 147,903; B., 1921, 776 a.

Apparatus for the separation of iron and slag. W. Diedrich (E.P. 267,008, 4.9.26).

Separation of mineral pulp (E.P. 264,129).—See I.

XI.—ELECTROTECHNICS.

Electric furnace with revolving arc. G. E. Evréinoff and S. Y. Telny (Rev. Mét., 1927, 24, 57—63).—An arrangement for producing a revolving arc with direct current consists of a carbon electrode with a graphite pot, in which the charge is melted, as positive electrode. A coil, in series with the arc, surrounds the pot. The electrode *E.M.F.* varies linearly with current strength in the surrounding coil and with the diameter of the pot. The effect of furnace temperature on the *E.M.F.* is investigated. A corresponding arrangement for alternating current is described. Advantages of a revolving over a fixed arc are the greater volume of arc and greater heat-radiating surface in the former case with consequent diminution of local overheating and longer life of refractory linings in the furnace. L. M. Clark.

Transformer iron and wattage losses. Eichenberg and Oertel.—See X.

Potential of zinc–copper alloys. Bauer and Vollenbruck.—See X.

Patents.

Galvanic cell. A. Heil (G.P. 435,747, 15.5.25).—In an electric cell an alloy of chromium, preferably containing cobalt and nickel, and if desired manganese, is used as the positive electrode. With such an electrode, a cell of size 27 × 22 × 10 cm. will afford a current of 10 amp. for 15—20 hrs. J. S. G. Thomas.

Electric cell with constant intensity. J. Pellini (U.S.P. 1,621,518, 22.3.27. Appl., 30.6.25. Conv., 22.11.24).—An outer vessel forms the negative element, and is separated by a porous tubular member from a centrally arranged hollow perforated carbon member forming the positive element. The electrolyte placed in the outer vessel consists of an aqueous solution of magnesium sulphate, potassium sulphate, and sodium sulphate, whilst the depolarising liquid contained within the porous member is composed of a slightly acidified aqueous solution of sodium bichromate. The relative proportions of electrolyte and depolarising liquid are chosen so as to maintain a constant voltage of the cell for a predetermined time. J. S. G. Thomas.

Electrolytic decomposing cell. F. Lawaczeck (E.P. 266,803, 30.11.25).—The opposed faces of pairs of closely packed, perforated, co-operating electrodes are electrically insulated from one another by enamel, rubber, varnish, etc., so that gases are evolved during electrolysis only from surfaces behind the plane of opposed faces. A perforated member, *e.g.*, of hard rubber, may be interposed between opposed electrodes, which are clamped together. J. S. G. Thomas.

Recovery of the filling material of used dry batteries. Chem. Fabr. Johannisthal G.m.b.H., K. von Vietinghoff-Scheel, and F. Trostler (G. P. 435,748, 7.1.26).—The material is extracted with dilute acids at ordinary temperature and then treated with an aqueous solution of sulphur dioxide at a higher temperature. Finally, recovered graphite is treated with stronger acids in the presence of sulphur dioxide for removal of iron. J. S. G. Thomas.

Manufacture of luminous electrical [neon-] discharge tubes. R. R. Machlett (U.S.P. 1,618,767, 22.2.27. Appl., 24.9.26).—The performance of neon-filled discharge tubes is improved if in their manufacture they are evacuated and heated, then filled with potassium vapour, and a high-frequency current passed through them. Removal of occluded gas is complete. T. S. Wheeler.

Electric insulators. Österreichische Siemens-Schuckert-Werke (Austr. P. 104,007, 4.10.23).—Rubber latex treated with vulcanising substances, dyes, fillers, etc. is applied to articles to be covered with insulating material, and allowed to dry. J. S. G. Thomas.

Making magnetic cores. W. Ehlers, Assr. to General Electric Co. (U.S.P. 1,618,818, 22.2.27. Appl., 29.12.24. Conv., 29.1.24).—Magnetic cores for induction coils and the like are prepared by pressing a mixture of pulverised, magnetic material, and a relatively infusible, insulating substance, *e.g.*, porcelain or magnesia, at a temperature above that at which the magnetic material softens, but below the m.p. of the insulating substance. Alternatively a relatively fusible insulating material is employed. T. S. Wheeler.

[Preventing deformation of electrodes in] electric accumulators. C. H. O. Lübeck (E.P. 242,290. 28.10.25. Conv., 31.10.24).

Dry battery. Burgess Battery Co., Assees. of W. B. Schulte (E.P. 245,464, 1.1.26. Conv., 2.1.25).

[Separator for] electric storage batteries. Fool-Pruf Patent Accumulator Co., Ltd., and F. G. Brown (E.P. 266,797, 26.11.25).

Electrical treatment of gases etc. (E.P. 266,983).—See I.

Treatment of hydrogen sulphide in gases (E.P. 267,018).—See VII.

XII.—FATS; OILS; WAXES.

Improvement in the quality of soaps made with hydrogenated oils. I. M. Hirose (J. Soc. Chem. Ind. Japan, 1927, **30**, 122—128).—The effect of the addition of castor oil soap to the soap of hydrogenated herring oil is studied. A slight decrease in the drop number is observed, but the lathering power increases remarkably even by the addition of 5% of the soap, and reaches a maximum when 20% has been added. The washing power measured by the indigo method is only slightly affected by the addition of a small amount of castor oil soap, but when more than 15% is added the washing power decreases gradually. The compound soap made with castor oil fatty acid (2—3 pts.), herring oil soap (90 pts.), and castor oil soap (10 pts.) gives very good results both in the washing and lathering power. K. Kashima.

Spontaneous heating of oils. Methods of testing. N. J. Thompson (Ind. Eng. Chem., 1927, **19**, 394—397).—Clean cotton waste (30 g.) impregnated with the oil to be tested (30 g.) is placed in a horizontal oil-jacketed steel tube allowing access of air to the material. The temperature is maintained at 104·4° until the temperature of the sample exceeds that of the bath by 2°; the temperature of the bath is then raised at the rate necessary just to maintain this difference. The relative tendency of an oil to heat spontaneously is shown by the temperature–time curve so obtained. Contrary to results previously recorded, the heating rate of lard oil is greater than that of cottonseed oil, the erroneous values given by the earlier methods being due possibly to the different accelerating effects of the products of partial oxidation in the two cases. A modified Mackey method using a larger sample (30 g. of oil on 30 g. of cotton) gives results comparable with those of the apparatus here described. In determining the hazard due to the spontaneous heating of oils the test method should be carried out under conditions similar to those under which the oil is to be used. If the presence of a retarding agent is suspected the duration of the test should be suitably extended. A. B. Manning.

Application of thermal analysis [to waxes]. K. Arndt (Z. angew. Chem., 1927, **40**, 314—316).—Thermal analysis may be applied to the study of waxes used in electroplating etc. When cooling curves of these waxes are plotted they show a number of breaks in addition to the first one at the m.p. If, instead of temperature, the rate of cooling is plotted against time, there are strongly marked minima at the temperatures at which the breaks in the original curves occur. By mixing two waxes the minima characteristic of both are reproduced, but at somewhat lower temperatures. The exact positions of the minima vary to some extent with the treatment to which the wax has been subjected; thus, *e.g.*, for wax which has been partly saponified, a much higher m.p. is obtained. Other possible applications are indicated. M. S. Burr.

Action of fatty acids on rubber. Smith and Boone.—See XIV.

Patents.

Manufacture of finely-divided dry soap. R. L. Holliday, Assr. to Industrial Spray-Drying Corp. (U.S.P. 1,621,506, 22.3.27. Appl., 19.4.26).—Freely-flowing liquid soap stock is sprayed into a current of heated gas, in which the progressively drying soap particles remain in suspension until their moisture content is reduced to the desired value. S. S. Woolf.

Production of vitamin preparations. A. W. Owe (E.P. 266,905, 2.3.26).—Vitamin-bearing marine fats are saponified by means of an alkali or alkaline-earth substantially free from water. The soap thus formed is extracted in a practically undissociated condition by lixiviation with a liquid edible fat, in the absence of light and oxygen. S. S. Woolf.

Manufacture of solutions [of fats etc.]. I. G. Farbenind. A.-G., Assees. of Chem. Fabr. vorm. Weiler-

ter Meer (E.P. 245,129, 21.12.25. Conv., 22.12.24).—Waxes and fats and any admixed dyestuff are dissolved in a solution containing a monoalkyl ether of ethylene glycol, propylene glycol, or butylene glycol.

B. Fullman.

Production of highly oxidised oil suitable for use as emulsifying agent, *e.g.*, in the manufacture of margarine. K. H. Hansen (G.P. 396,426, 13.3.23). —A mixture of oil with a high iodine value with oil having an iodine value of less than 10 is treated with air or oxygen at about 190°, but below 200°, until the unsaturated oil is oxidised sufficiently. A small quantity of calcium hydroxide may be added to the product to prevent it from adhering to the walls of the apparatus.

L. A. Coles.

Production of soaps and detergents. Colloidal Products Co., Assees. of R. M. Pettit (E.P. 243,735, 25.11.25. Conv., 27.2.25).—See U.S.P. 1,544,103—4; B., 1925, 680.

Process for separating volatile substances. E. Wecker (U.S.P. 1,622,126, 22.3.27. Appl., 13.3.24. Conv., 20.3.23).—See G.P. 397,332; B., 1924, 877.

Preparation of sulpho-aromatic fatty acids (E.P. 252,212).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

[Paint] driers. H. Wolff (Farben-Ztg., 1927, **32**, 1490—1491).—The tensile strength and elasticity of dry detached films of paints made by adding linoleates or resinates of cobalt, manganese, or each of these metals in conjunction with cerium, to a paint base consisting of 68·0% of lithopone, 0·5% of lampblack, and 31·5% of linseed oil, indicate that the influence of driers on paints etc. must be considered not only from the point of view of induced drying time, but also from that of effect on mechanical properties of the film. The author stresses the significance of the colloid-chemical reactions caused by a drier rather than its chemical composition or metal content. Differences in the degree of dispersion of the metal account for the varying results of substituting resinate for linoleate driers, and for the non-concordant mechanical properties of films made with driers of identical chemical composition, but coming from different sources. The use of cerium in conjunction with cobalt or manganese is recommended.

S. S. Woolf.

Oil absorption of lithopone. E. Klumpp (Farben-Ztg., 1927, **32**, 1491—1492).—A general discussion on lithopones of high and low oil absorption and their suitability for various purposes. The opacity of paints based on lithopone of low oil absorption is gained at the expense of durability, whereas lithopone paints of high oil absorption are durable, but have relatively poor hiding power.

S. S. Woolf.

Patents.

Manufacture of colour lakes. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 265,032, 29.3.26).—A basic dye, whether containing acid groups or not, is treated with components capable of forming complex tungstic acids (except those containing molybdenum) in the presence of an acid more highly dissociated than tartaric acid, such as hydrochloric, sulphuric, nitric, oxalic, trichloroacetic, or formic acids, such acid not taking part (as does phosphoric acid) in the reaction. The colour lakes may be precipitated in the presence or absence of the usual substrates.

A. Davidson.

Manufacture of solutions [for varnishes etc.]. I. G. Farbenind. A.-G., Assees. of Chem. Fabr. vorm. Weiler-ter Meer (E.P. 252,203, 21.12.25. Conv., 22.12.24).—Resins, cellulose esters, and any admixed dyestuff are dissolved in a solution containing a substantial proportion of a monoalkyl ether of ethylene glycol, propylene glycol, or butylene glycol, with the exception of the monoethyl compound. B. Fullman.

Synthetic resin and its manufacture. E. R. Littmann, Assr. to Commercial Solvents Corp. (U.S.P. 1,618,209, 22.2.27. Appl., 25.11.25).—Sodium ethyl, butyl, or other alkyl 3- or 4-nitrophthalate, is treated in solution with copper sulphate, or ferric chloride, or other soluble salt of a heavy metal, when the corresponding heavy metal salt of the alkyl hydrogen nitrophthalate is obtained, in the form of a resinous substance of value as a substitute for natural resins.

T. S. Wheeler.

Composition and processes for coating or filling surfaces. F. R., M., and A. Hervé (E.P. 253,889, 7.6.26. Conv., 16.6.25).—See U.S.P. 1,615,584; B., 1927, 259.

Luminescent product. A. A. Guntz (U.S.P. 1,622,379, 29.3.27. Appl., 8.5.22. Conv., 10.6.21).—See E.P. 181,333; B., 1923, 191 A.

Production of titanium pigments from materials containing titanium. E. C. R. Marks. From R. H. Monk and J. Irwin (E.P. 266,211, 6.8.26).—See U.S.P. 1,542,350; B., 1925, 638.

Manufacture of varnish. F. Schwartz, Assr. to E. Gil-Camporro (U.S.P. 1,621,438, 15.3.27. Appl., 19.3.25. Conv., 26.3.24).—See E.P. 231,457; B., 1926, 373.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Röntgenographic studies with metastyrene. (Miss) M. Hünemörder (Kautschuk, 1927, 106—107).—Between 80° and 105° metastyrene resembles rubber in its elasticity. By repeatedly warming and stretching it is possible to obtain an extension of 3000—4000%. In this condition metastyrene gives an interference *X*-ray diagram distinctly different from that of the unstretched material, and indicative of incipient orientation of the particles. Extrusion through fine apertures fails to induce any more marked orientation. The behaviour in this respect recalls that of synthetic rubber.

D. F. Twiss.

Alternating behaviour of fatty acids added to rubber compounds. W. H. Smith and C. E. Boone (Ind. Eng. Chem., 1927, **19**, 398—399).—Using a mixture of rubber (100 pts.), sulphur (7—10 pts.), zinc oxide (5 pts.), and fatty acid (1/150 mol.), the tensile strength of the vulcanised product shows an alternating relationship as the homologous series of fatty acids is ascended from formic acid to stearic acid; the curve for the tensile strength of the products containing the acids with an even number of carbon

atoms lies above that for the acids with an odd number. There is no relation between the number of carbon atoms and the final elongation. With the phenyl-substituted fatty acids there is progressive acceleration from benzoic acid to hydrocinnamic acid.

D. F. Twiss.

Magnesium carbonate and the transparency of vulcanised rubber. Y. Tanaka (Trans. Inst. Rubber Ind., 1927, **2**, 330—344).—Magnesium carbonate, which is used extensively in Japan in the production of transparent rubber goods, varies considerably in its quality for this purpose. Examination of the behaviour of a number of commercial samples shows that the accelerating power and the degree of beneficial effect on the mechanical properties lie in the inverse order to the transparency in rubber. The more transparent grades consist of amorphous particles, whereas those consisting entirely or in part of needle crystals are inferior in this respect. The former have a refractive index approximately equal to that of vulcanised rubber, whilst the latter contain a mixture of particles of diverse refractivities. Satisfactory magnesium carbonate for the production of vulcanised rubber can be selected by examination of the effect on the appearance of a mixture of nitrobenzene and petroleum adjusted to n_D 1·525, or of Canada balsam (n_D 1·52—1·53), or of cedar oil (n_D 1·51—1·52). D. F. Twiss.

Volatile oxidation product of balata. T. R. Dawson and B. D. Porritt (Trans. Inst. Rubber Ind., 1927, **2**, 345—353).—Venezuelan sheet balata purified by extraction with acetone and separation from clear solution in benzene by the addition of alcohol was exposed to a current of air at the ordinary temperature. The oxidation process followed an auto-catalytic course, and yielded a final product approximating to the composition $C_{10}H_{16}O_3$. A small quantity of formic acid also was produced, but no carbon dioxide; on the assumption that one carbon atom in every $(C_{10}H_{16})_n$ is converted into formic acid the value of n would be 112.

D. F. Twiss.

Ageing properties of raw and vulcanised rubber. G. Martin (Trans. Inst. Rubber Ind., 1927, **2**, 354—380). —Examination of mixtures with sulphur (90 : 10) vulcanised to give an elongation of 860% at a load of 1·04 kg./sq. mm., shows no difference in the "ageing" properties of quickly-dried and "matured" rubber from comparable coagula. Sheet rubber, however, ages better than crêpe rubber. Sheet rubber also can be vulcanised over a much wider range than crepe without the development of rapid deterioration in the tensile strength on ageing. With respect to maintenance and tensile strength, evaporated latex is superior to sheet and hard fine Para rubber, and still more so to crêpe rubber; on the other hand, with respect to extension under a given load, hard fine Para undergoes less change than crêpe or sheet rubbers, and these less than evaporated latex. These differences are attributable to the serum substances, the removal or addition of which is shown to have a marked effect. Treatment of rubber with aqueous ammonia removes substances responsible for its satisfactory ageing. After extraction with acetone likewise the ageing properties of rubber are depreciated, but the addition of serum solids in this case has little beneficial effect, although the re-introduction of the acetone extract is very effective. It is probable, therefore, that more than one substance is necessary for the satisfactory ageing properties of plantation rubber, and that a particular balance of these active non-caoutchouc constituents is of great importance.

D. F. Twiss.

Rubber solvents. D. F. Twiss (Trans. Inst. Rubber Ind., 1927, **2**, 381—408).—The more important solvents for rubber are reviewed, and the relative behaviour of rubber towards them is considered, *e.g.*, as to rate of swelling or viscosity and transparency of the resulting solutions. Other features of practical importance such as vapour pressure, relative inflammability, and solubility for water and sulphur are also discussed.

D. F. Twiss.

Rubber content of ammoniated latex. R. O. Bishop (Malayan Agric. J., 1927, **15**, 1—11).—The apparent rubber content of latex preserved with a minimum quantity of ammonia, as indicated by evaporation, or coagulation with acetic acid or alcohol, rises slightly during the early stages of storage and then gradually decreases. The weight of coagulum with acetic acid also varies with the concentration of the acid coagulant and with the proportion of acid in excess of that required for the neutralisation of any ammonia present. Accurate evaluation of the rubber content of latex by coagulation with acetic acid is consequently not simple, and for the ensuring of concordant results the method must be elaborated. D. F. Twiss.

Patents.

Manufacture of rubber-compounding material. Barrett Co., Assees. of A. B. Cowdery (E.P. 243,384. 21.11.25. Conv., 22.11.24).—Coal tar is distilled so as to give a residue containing approximately 60% of finely-divided carbon and 40% of hydrocarbons. This product, having d 1·30—1·35 and m.p. above 177°, is used as a compounding ingredient for rubber.

D. F. Twiss.

Manufacture of rubber. P. Schidrowitz, and Vultex, Ltd. (E.P. 266,418, 28.10.25. Addn. to E.P. 193,451; B., 1923, 732 a).—Concentrated latex produced by any convenient process is mixed with vulcanising ingredients, *e.g.*, sulphur or polysulphides, accelerators, and with other compounding ingredients, suitable preservatives and preservative colloids, if necessary, also being present. Vulcanisation may be effected by a low-temperature process or under the more customary conditions above 100°. D. F. Twiss.

Electric insulators (Austr. P. 104,007).—See XI.

Biguanides [as accelerators] (G.P. 435,668).—See XX.

XV.—LEATHER; GLUE.

Mechanism of tanning. I. Fixation of vegetable tannins by chrome-tanned hide protein. K. H. Gustavson (J. Amer. Leather Chem. Assoc., 1927, **22**, 125—139).—Samples of hide powder treated with anionic, cationic, and mixed chromium compounds respectively, were then subjected to a solution of hemlock bark extract, gallotannic acid, or sulphited quebracho extract. The cationic chrome-tanned powder had a much greater affinity for the vegetable tannins than

either the anionic chrome-tanned powder or the original powder in the usual p_H range for vegetable tanning, viz. 3—6. Untanned hide powder fixed more tannin than anionic chrome-tanned powder except at high p_H values. The acidity of the chrome–collagen compound was greatly reduced by the vegetable tannage. The portion of the acid combined with the collagen was completely removed by the vegetable retannage, and acido-groups were also removed from the chromium complex. The latter was accompanied by a loss of fixed chromium. Hide powder freshly tanned with cationic chromium compounds loses chromium when treated with vegetable tannins, but if it is "aged" before the latter treatment there is no loss of chromium. Anionic chrome-tanned powder loses chromium with vegetable retanning, whether "aged" or not. This supports the view that the same protein groups are affected by anionic chromium compounds and vegetable tannins. The increased capacity of cationic chrome-tanned powder for vegetable tannin is attributed to the activation of the basic protein groups, resulting from the breaking up of the closed structure by the reaction of acidic protein groups with chromium complexes. The fixation of vegetable tannin by chrome-tanned powder was practically independent of the p_H value of the vegetable tannin solution. The Procter–Wilson theory was not applicable in this case. This is attributed to the rigid structure of the chrome-tanned powder. The effects of hydrogen and hydroxyl ions are negligible compared with those on collagen itself.

D. Woodroffe.

Action of trypsin on calfskin. H. B. Merrill and J. W. Fleming (J. Amer. Leather Chem. Assoc., 1927, **22**, 139—161).—Experiments have shown that all the collagen in calfskin is capable of digestion by trypsin. The rate of digestion increases considerably between 35° and 40°. It is a maximum on the alkaline side of p_H 8·0. Under usual conditions of bating, the action of the trypsin on the collagen is so slight that little or none is lost during that operation. The non-collagen proteins of limed skin constitute 5% of the dry weight of the skin. The quantity of material removed in a few hours from limed calfskin by trypsin increases with the duration of the preceding liming. Marriott's theory of "altered collagen" (B., 1926, 682) and the "protective sheath" theory (B., 1923, 367 A) are discussed. It is shown that the type of curve obtained from the rate of hydrolysis of collagen by trypsin does not agree with these theories, but suggests that the collagen is equally susceptible to the action of the trypsin. The principal function of trypsin in the bating of calfskins is the removal of protein degradation products formed in the lime liquor, of which the most important is probably keratose, the constituent of the interfibrillary substance or solution. The longer the liming, the greater the amount of degradation products to be removed. No elastin is removed from calfskin during the ordinary bating operation. It is probable that its removal is more important in goatskin bating. Keratose is present in limed skins, and is digested by trypsin, so that it is probably removed by bating. Keratose is precipitated by solutions of p_H less than 4·1, and it seems probable that the precipitation of such in a skin would impair the finished leather.

D. Woodroffe.

Bating action and methods of measuring enzymic activity of natural and artificial bates. G. Hugonin (J. Soc. Leather Trades' Chem., 1927, **11**, 1—18).—The characteristics of a bated skin are the cleansing of the fibres from interfibrillary substance and from degraded collagen produced in the liming, the "fallen" supple nature, the absence of elasticity and swelling, the porosity to air, the scud is easily raised by scraping the grain, the latter has a feel like oily glass, and most of the lime is removed. The activity of the enzymes present in bates is affected by the temperature of the medium, the enzyme concentration, the deliming agent, neutral salts, hydrogen-ion concentration, and suspended matter which can absorb the enzymes and increase their activity. Pepsin has optimum action at p_H 1·4, does not readily dissolve the elastin, and attacks the grain and flesh sides equally. The optimum effect of trypsin is at p_H 8·0. The liquor should be alkaline to cresol red and acid to phenolphthalein. Tryptic hydrolysis is more drastic than peptic digestion. It hydrolyses proteins to simple polypeptides and amino-acids. Blood serum stimulates tryptic action. Fresh pancreatic extract has only feeble proteolytic activity, but the latter increases after a while and under certain conditions. The pancreas of animals slaughtered in course of digestion becomes active more readily than if slaughtered fasting. The pancreatic extracts of dogs and cattle activate equally well, that of cats only with difficulty. Extraction with boiling water or glycerin water renders it inactive. Unboiled water or water acidified with acetic or lactic acids furnishes an active extract. The addition of amino-acids renders it still more active. The activating agent is enterokinase. Pancreatic trypsin differs in its action from pepsin by forming several compounds titratable with formaldehyde (amino-acids, tyrosine, tryptophan, etc.), by showing a tendency in weak solutions to build up synthetic products, and by not reaching the limiting amount of peptone by a gradual process, but in a series of oscillations. Trypsin acts in liming by dissolving the keratinous layers in contact with the hyaline layer and in bating, where it digests the elastic fibres and the keratose and greases without attacking the collagen. Erepsin is obtained from the small intestine of dogs. It does not act on natural proteins, but acts on albumoses and peptones, and its optimum effect is the same as for trypsin. Pancreatin always contains erepsin. Bating "falls" the skin by acting as a "buffer" of p_H 8·0. Boric acid, ammonium sulphate and chloride, weak organic acids and substances which form them by fermentation, and sodium dihydrogen phosphate may each be used for deliming and "falling" of pelts. Bating usually removes lime, but some artificial bates contain insufficient deliming agent. A preliminary controlled acid deliming is advisable before bating. The pancreatic enzymes are capable of dissolving the elastin fibres during bating, and this property has been suggested for purposes of controlling the digestive power of the enzymes. The speed of digestion of elastin by the enzymes is proportional to the concentration and the time of action. Ammonium chloride (0·5 g./litre) stimulates the enzyme activity at p_H 7·6. Larger quantities have the opposite effect. Solutions of greater concentration than 5% hydrolyse the collagen fibres. Most of the hide substance

dissolved during bating is due to the previous action of the lime liquors on the pelt. Methods have been devised for controlling the effects of bating.

D. Woodroffe.

Leather dyeing. VI. H. Salt (J. Soc. Leather Trades' Chem., 1927, **11**, 18—19).—Sheep grains tanned by the one-bath and two-bath chrome tannages, respectively, were dyed with a number of acid, basic, and direct dyes, respectively. The acid and direct dyes produced full shades on both types of leather and the basic dyes weak shades. The two tannages are apparently chemically alike with respect to dyeing properties.

D. Woodroffe.

***X*-Ray examination of the tanning of membranes and tendons.** R. O. Herzog (Kolloid-Z., 1927, **41**, 277).—Polemical (see Katz and Gerngross; B., 1927, 150).

E. S. Hedges.

Patents.

Apparatus for drying leather (E.P. 266,464).—See V.

Dyeing leather (E.P. 247,187).—See VI.

XVI.—AGRICULTURE.

Reversion of nitrates in the soil under cultural conditions in Mauritius. N. Craig and F. Giraud (Dept. Agric. Mauritius, 1926, Bull. No. 11, 19 pp.).—Laboratory experiments are reported on the extent to which denitrification takes place when organic manures are applied to soil at the same time as ammonium sulphate or sodium nitrate. Addition of large amounts of molasses to a soil in which nitrification normally proceeded fairly rapidly, inhibited nitrification and produced conditions under which ammonical and nitric nitrogen reverted to organic nitrogen. Dried green manure and farmyard manure acted in the same manner, but to a somewhat less extent. Molasses and green manure did not cause an increase in the loss of nitrogen as gas, whereas with farmyard manure a large increase in this loss occurred. The results support and explain the conclusions drawn from previous field experiments with sugar cane on Mauritius soils.

C. T. Gimingham.

Relation between the "citric soluble" and "root soluble" nutrients in soils. G. Hasenbäumer and R. Balks (Z. Pflanz. Düng., 1927, **B 6**, 116—122).—Additions of phosphoric acid, as basic slag or superphosphate, to soil in various proportions, and subsequent extraction with 1% citric acid, gave average figures of 98% of the added P_2O_5 recovered in the case of light soils, and 65% in the case of heavy soils. Corresponding figures for the amounts of added P_2O_5 recovered in rye seedlings, used according to Neubauer's method for determination of available plant nutrients in soils, were 35% and 31% for light and heavy soils respectively. With added potassium, on the other hand, an average of 82% was recovered in the seedlings and 69% by extraction with citric acid.

C. T. Gimingham.

Influence of drying and heating arable soils on their content of water-soluble phosphoric acid. A. T. Schlœsing and D. Ledoux (Compt. rend., 1927, **184**, 649—652).—Samples of soil, dried by sun or in an oven, and undried, were washed under standard conditions with water containing 0·08 g. of calcium nitrate per litre. The washings were repeated until determinations of phosphoric acid gave the same value on washings coming from the dried and the untreated soil. Lebediantzeff's results (B., 1924, 305, 346) have been confirmed on soil from Dangu (dept. Eure) and Villepreux (Seine-et-Oise) taken at depths of 10—25 cm. and 15—25 cm. respectively, but the above method shows differences in the water-soluble phosphoric acid content between the dried and untreated soil about 50 times greater than those found by Lebediantzeff.

R. Brightman.

Relative weights of reacting substances in colloidal flocculations. J. Dumont (Compt. rend., 1927, **184**, 764—766).—Experiments on the flocculating power of 0·1*N*-solutions of ammonium, sodium, and potassium chlorides, and 0·01*N*-calcium and magnesium chlorides on different specimens of clay soils, before and after separation of the colloidal matter by Schlœsing's method (B., 1926, 717), show that the ratio of the weight of flocculant actually participating in the reaction to the weight of gel formed varies with the sample of soil examined and increases with the colloidal content of the sample, though without definite relation to the ratio of slime to colloids in the soil. The limiting amounts of flocculant required are not strictly proportional to the physical composition of the clay, although dependent on the nature and source of the latter. The clay constituents of soils represent either a mixture of electronegative colloids tending to disperse, or a colloidal complex with amphoteric tendencies which has resulted from the combination of colloids with opposite charges.

R. Brightman.

Manganese deficiency in soils and fertilisers. O. Schreiner and P. R. Dawson (Ind. Eng. Chem., 1927, **19**, 400—404).—A description of laboratory and field experiments on the growing of tomatoes in a highly calcareous and slightly alkaline soil (82—92% $CaCO_3$, 5% of organic matter, manganese under 0·001%) occurring in Florida, which, with heavy application of inorganic fertilisers, failed to produce a crop unless stable manure was applied to the young plants. With fertilisers alone and without manure the plants failed to blossom, and developed a strikingly characteristic chlorosis indicative of a failure of normal leaf function caused by inadequate chlorophyll synthesis. The addition of from 25—50 p.p.m. of manganese sulphate together with the application of a balanced inorganic fertiliser or peat caused a strong improvement in the growth, colour, and yield of fruit, the plants manifesting a luxuriant growth from the start. Under the conditions prevailing in this type of soil the presence of manganese is indispensable to the normal growth of the tomato plant.

E. H. Sharples.

Manuring of chicory. J. Nuding (Z. Pflanz. Düng., 1927, **B 6**, 97—115).—Details are given of manurial experiments with chicory, and the results are discussed from an economic point of view.

C. T. Gimingham.

Sulphite liquor as a spray. Fleming and Reedy.—See V.

Patents.

Measurement of soil moisture. Siemens & Halske A.-G., Assees. of O. Werner (G.P. 435,229

6.3.25).—A rod- or band-shaped device is made of a porous substance, *e.g.*, blotting paper, and inserted in the soil to a determined depth. From the time taken for the moisture absorbed from the soil to reach a certain height above the surface a measure is obtained of the moisture content of the soil. G. W. ROBINSON.

Apparatus for measurement of suction force and moisture content of soils. W. KORNEFF (G.P. 435,815, 1.4.25).—A porous vessel is connected with a non-porous vessel furnished with a thermometer and manometer. The whole apparatus is filled with water. The porous vessel is plunged in the soil and the variations as shown by the manometer give a measure of the changes in the moisture content of the soil.
G. W. ROBINSON.

Stabilisation of hydrocyanic acid for use as an insecticide. CHEM. FABR. DR. H. STOLTZENBERG (G.P. 435,714, 3.7.25).—Sulphur dioxide is pumped into a steel receptacle maintained at a low temperature and containing liquid hydrogen cyanide, if necessary under pressure. The receptacle is allowed to attain ordinary temperature. It now contains liquid sulphur dioxide with an admixture of hydrogen cyanide as a liquid phase, and a mixture of the two gases as the gaseous phase. The pressure is used to force out the liquid through a tube, which reaches to the bottom of the receptacle, and is furnished with an atomising jet to produce a fine spray or cloud which rapidly vaporises.
G. W. ROBINSON.

Insecticidal plant spray. I. G. FARBENIND. A.-G., Assees. of R. MAY and W. MISCHON (G.P. 435,713, 4.3.23).—The first runnings from benzene distillation, with or without addition of emulsifying agents, have an insecticidal action greater than that corresponding with the amount of carbon disulphide present, and are without the disadvantages of carbon disulphide. The insecticidal effect may be due to constituents such as thiophen, alcohols, and hydrocarbons. G. W. ROBINSON.

[Increasing adhesiveness of] insecticidal and like powders. I. G. FARBENIND. A.-G., Assees. of FARBENFABR. VORM. F. BAYER & Co. (E.P. 250,180, 21.1.26. Conv., 3.4.25).—The adhesive properties of insecticidal and fungicidal dusts are greatly increased by intimate admixture with small quantities of a product obtained by treating alkali cellulose with an alkali salt of a halogen-substituted aliphatic acid. C. T. GIMINGHAM.

Insecticide and fungicide. K. S. BOYNTON, Assr. to E-Z-WAY Co. (U.S.P. 1,621,825, 22.3.27. Appl., 25.7.23). —Sodium sulphide is melted in its own water of crystallisation and sulphur is added to substantial saturation. The mixture of sodium polysulphides thus formed is allowed partly to cool, and is mixed with soap until a paste-like consistency is assumed. 2% of copper sulphate may also be added. C. T. GIMINGHAM.

Production of manure by treatment of organic refuse. A. BAUMGARTEN-CRUSIUS (E.P. 246,484, 21.1.26. Conv., 21.1.25).—Organic refuse is caused to undergo a process of hot fermentation by blowing air through the mass, stacked in loose layers. The mass is maintained above a minimum temperature of 50° (*e.g.*, at about 65°), which ensures destruction of weed seeds and harmful organisms, and causes the fermentation to be greatly accelerated. The resulting manure may be used at once, or stored under conditions which facilitate the initiation of a second fermentation by renewed aeration.
C. T. GIMINGHAM.

Manufacture of fertilisers. G. CLAUDE, Assr. to SOC. L'AIR LIQUIDE (SOC. ANON. POUR L'ETUDE ET L'EXPLOITATION DES PROCÉDÉS GEORGES CLAUDE) (U.S.P. 1,621,311, 15.3.27. Appl., 20.12.23. Conv., 24.1.23. Renewed 5.4.26).—See E.P. 210,399; B., 1924, 530.

XVII.—SUGARS; STARCHES; GUMS.

PATENTS.

Citric acid (E.P. 266,414—5 and G.P. 434,729).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Fermentation in candy and creams. PAINE and others.—See XIX.

PATENTS.

Preparation of citric acid. B. BLEYER (G.P. 434,729, 19.10.24).—To a 10—15% solution of sucrose, glucose, or maltose, with the addition of nutrient salts (ammonium nitrate, potassium phosphate, and magnesium sulphate), mineral acid or citric acid is added to give a p_H of 3·3—3·6; the whole is sterilised by heating and then seeded with *Aspergillus*, *Sterigmatocystis*, *Penicillium*, or other related organisms. Gentle warming assists the growth. When germination occurs and short mycellar threads appear, sterilised air is passed in, the liquid is gently stirred, respiratory carbon dioxide being at the same time removed, and acidification commences and continues. For *Aspergillus* the optimum temperature is 15—20° and the maximum yield of citric acid (60—75%) is reached in 5—8 days.
C. HOLLINS.

Production of citric acid. A. FERNBACH, J. L. YUILL, and ROWNTREE & Co., LTD (E.P. 266,414—5, 26.10.25).—Solutions of sugars and like substances or mixtures thereof with added nutrient materials are fermented by moulds or fungi, in particular aspergilli. Sterilisation of the medium by heat is avoided by the addition of a sufficient quantity of mineral or strong organic acid, so that the hydrogen-ion concentration of the medium is raised, prior to inoculation, to such a point that not only bacteria but yeast and the majority of other fungi are inhibited, whilst the selected organism still functions. The solution is incubated at 10—40°, and is brought as far as possible into contact with the mould. The sugars and fresh nutrients may be added continuously or intermittently with or without the withdrawal of the citric acid liquor. C. RANKEN.

Revivifying carbon used in purifying ethylene. Revivifying carbon. A. A. BACKHAUS, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,619,326—7, 1.3.27. Appl., 27.9.24).—See E.P. 240,126—7; B., 1925, 1007.

Manure from refuse (E.P. 246,484).—See XVI.

XIX.—FOODS.

Volatile constituents of foodstuffs. J. KÖNIG and W. SCHREIBER (Z. Unters. Lebensm., 1927, **53**, 1—44).—Earlier work has shown that among the substances volatilised when certain foods are heated as in cooking,

may be included carbon dioxide, mercaptan, hydrogen sulphide, aldehydes, and formic acid. By the authors' method all these compounds are determined in one operation by using ammoniacal silver solution as a common absorbent. The material under investigation is heated at 98—100° in an atmosphere of nitrogen for 2 hrs. on a paraffin bath. Four absorption vessels are used containing ammoniacal silver hydroxide solution (3 g. of silver oxide in 100 c.c. of 24% ammonia solution), the third vessel being warmed at 50° and the last serving as a control. Coloured deposits and silver mirrors are formed during the distillation. The contents of the receivers are filtered and systematically analysed. The combined sediment is treated with 5% hydrochloric acid to liberate mercaptan, and then with concentrated acid to liberate hydrogen sulphide, both gases being oxidised by alkaline hydrogen peroxide and determined as sulphuric acid. The residue is treated with ammonia and nitric acid, silver being then precipitated with hydrochloric acid, dried, and weighed. The filtrate after removal of silver is evaporated to dryness, dissolved in nitric acid, and analysed for phosphoric acid. The filtrate from the absorption vessels is boiled (in absence of air) with 3% baryta solution. The precipitate is filtered and decomposed with hydrochloric acid, the carbon dioxide evolved being absorbed in potash. The filtrate is evaporated to dryness with Devarda's alloy, the residue being dissolved in water, acidified with sulphuric acid, and distilled. The formic acid in the distillate is precipitated with mercurous chloride, filtered, dried, and weighed. The residue after distillation is extracted with ether, from which higher organic acids are deposited on evaporation. Details are given of the results obtained for a large number of animal and vegetable materials, the results for coconut oil being entirely negative. H. Dowden.

Sugar-tolerant yeasts in chocolate-coated creams. M. B. Church, H. S. Paine, and J. Hamilton (Ind. Eng. Chem., 1927, **19**, 353—357).—Cultural and microscopical examinations of chocolate-coated creams inoculated with purified yeasts obtained from commercial samples of "burst" chocolates have been made, and it is concluded that the bursting is primarily due to the formation of minute quantities of gas in the cream by the activity of yeasts of high sugar tolerance. The yeasts are of various types, and are either spore-forming or torula-like or a mixture of both types. Experiments on the inoculation of creams with various spore-forming anaerobes gave no evidence of sugar tolerance for these bacteria and no bursting occurred. E. H. Sharples.

Means for preventing "explosive" or bursting fermentation of chocolate-coated fondant candy. H. S. Paine, V. Birckner, and J. Hamilton (Ind. Eng. Chem., 1927, **19**, 358—363).—The fermentation of chocolate-coated creams made from syrups of different composition and consistency and inoculated with highly sugar-tolerant yeasts has been examined. The bursting caused by the presence of these organisms may be prevented by the addition of invertase. This, by inverting a portion of the sucrose present and thereby increasing the sugar solubility, causes an increase in the density and osmotic pressure of the syrup phase of the fondant, and renders it resistant to fermentation. Fondants containing a syrup phase with a solids concentration of 79% or over caused by the addition of invertase were free from fermentation. E. H. Sharples.

Pectins. A. Mehlitz (Kolloid-Z., 1927, **41**, 130—146).—A method is described for the fractional filtration of solutions of fruit pectins through membranes graded with respect to the size of the pores, and a description is given of experiments on the determination of the size of the particles in apple pectin by this method. About one third of the particles are greater than 0·6μ, about two thirds are between 0·2μ and 0·6, and less than 10% are smaller than 0·2μ. Apple pectin of p_H 3·00 on fractional filtration undergoes a decrease in titration acidity, hydrogen-ion concentration, and viscosity with decrease in the size of the pores of the filter, the change of acidity being affected by the duration of filtering. All fractions gave a positive starch reaction, which was weaker in those passed through the finer filters. A close parallelism was observed between the methoxyl content of the pectin fractions and their ability to gelatinise (cf. Nanji and Norman, B., 1926, 930). E. S. Hedges.

Patents.

Sterilising and preserving milk and similar liquids. M. Bing (G.P. 392,935, 11.5.22).—Fresh milk in a finely-divided state is subjected to the action of ultra-violet rays, treated with hydrogen peroxide, and the excess of the latter decomposed by addition of a small quantity of fresh milk or cream. The product is germ-free, and resembles natural milk in taste. F. R. Ennos.

Improvement of creams which have undergone certain fermentations, and production of butter. M. O'Callaghan (E.P. 266,951, 27.5.26).—The cream is neutralised by the addition of a solution of calcium hydroxide or sodium bicarbonate, whereby the nitrogenous matter is precipitated, and heated to the pasteurising temperature. While being centrifuged it is washed with a current of acidified water to remove the flavour due to the alkali and the nitrogenous matter, and then mixed with a current of water or sweet milk, according as it is desired to manufacture butter or reconstituted cream. F. R. Ennos.

Extraction of albumin and nutritive salts from the water of potatoes. R. Vogel (G.P. 400,414, 13.1.21).—The water is made to froth by whisking or stirring, and the foam skimmed off and dried. F. R. Ennos.

Renewal of used brine lake by extraction of its albumin. Gräff & Co. (G.P. 394,993, 19.7.22).—The lake is acidified with hydrochloric acid, the albumin which separates filtered in the usual way, and the filtrate neutralised with sodium hydroxide or carbonate solution. After re-filtering, if necessary, the lake is again ready for use. F. R. Ennos.

Treatment of miller's wheat to eliminate the wild garlic it contains. L. G. Patrouilleau (E.P. 260,283, 21.10.26. Conv., 22.10.25).—See F.P. 604,950; B., 1926, 848.

Mixed salt for nutritive purposes (G.P. 398,093).—See VII.

Vitamin preparations (E.P. 266,905).—See XII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Furfuraldehyde manufacture from oat hulls. I. The liquid-solid ratio. H. J. BROWNLEE (Ind. Eng. Chem., 1927, **19**, 422—424; cf. B., 1925, 114).—Experiments are described on the yields of furfuraldehyde obtained by steam-distillation of ground oat hulls digested with dilute sulphuric acid under a pressure of 60 lb. (153°) for 5 hrs. in a rotary digester. The acid used was equal to 2·1% by wt. of the hulls and the rate of distillation averaged about 1600 lb. of distillate per hr. from 4500 lb. of hulls. The furfuraldehyde yields in the distillate reached a maximum (10·3%, concentration 5·8%) with a liquid–solid ratio in the initial charge of 0·27 : 1. These values declined as the liquid–solid ratio was either increased or decreased. The total furfuraldehyde in the system, *i.e.*, yield in distillate and that obtained from the residue, was at a maximum (12·6%) at a ratio of 0·35 : 1. Steam requirements are discussed, and it is concluded that both from the point of view of increased yields and decreased steam consumption a low initial liquid–solid ratio is desirable. E. H. SHARPLES.

Effect of polarised light on the pharmacodynamic properties of some drugs. D. I. MACHT and J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1927, **16**, 106—110).—The action of polarised light of the visible spectrum on tincture of digitalis and on solutions of quinine and cocaine has been examined. The solutions were irradiated simultaneously with polarised and non-polarised light of the same intensity, and even comparatively short exposures (45—60 min.) to polarised light produced a distinct deterioration (10% or more) in their pharmacological activity. This change was not produced to the same degree even by longer exposures to ordinary non-polarised light. The methods used in assaying the solutions are described and the two new methods for testing the pharmacological activity of cocaine are outlined. E. H. SHARPLES.

Effect of ultra-violet and polarised light on mercurochrome. D. I. MACHT and J. H. HILL (J. Amer. Pharm. Assoc., 1927, **16**, 110-113).—Exposure of solutions of mercurochrome-220 (1 in 500) to ultra-violet rays ($\lambda = 1850$ Å. and upwards) or to polarised light produces no deterioration in the antiseptic or germicidal efficiency tested on cultures of *B. coli.* E. H. SHARPLES.

Colorimetric assay of strophanthus. L. W. ROWE (J. Amer. Pharm. Assoc., 1927, **16**, 113—115).—A series of comparative tests on the assay of tincture of strophanthus by the picric acid colorimetric method based on the Baljet reaction (A., 1919, ii, 438) and the Houghton frog method. The total activity being apparently always proportional, though not equal, to the alimentary absorbable activity, if a purification method is not necessary, the colorimetric method, using ouabain, 1 in 25,000, or tincture of strophanthus, U.S.P. X., 1 in 140, as standards, can be used as a preliminary test to a short final check of the activity by the frog method. The colorimetric method is not suitable for the assay of squill, convallaria, or veratrum preparations as judged by one attempt on each. E. H. SHARPLES.

Some constituents of Spanish moss. A. W. SCHORGER (Ind. Eng. Chem., 1927, **19**, 409—411).—Spanish moss (*Tillandsia usneoides*) contains galactan (less than 1%), araban, xylan, cellulose, and apparently a glucoside. The analyses of the dry material before and after retting, respectively, are: ash 5·51%, 0·54%; nitrogen 1·11%, 0·80%; pentosans 15·68%, 18·13%; non-carbohydrates 16·63%, 19·51%; methoxyl 1·86%, 3·03%; cellulose 46·78%, 38·87%; pentosans in cellulose —, 16·52%. Protein, chlorophyll, a carotinoid pigment, a sterol, and considerable quantities of a wax (saponif. value 41·24) are present in the non-carbohydrates. Methylpentosans and pectin are absent. E. H. SHARPLES.

Effect of vacuum distillation on vanilla extract. C. B. GNADINGER (Ind. Eng. Chem., 1927, **19**, 342—344).—Vanilla extracts concentrated under reduced pressures at temperatures below 60° showed a loss in quality, strength of flavour, and aroma varying from 10 to 40%, depending on the degree of concentration and the kind of bean. This was due to loss of volatile oils and acids, and not to chemical change or decomposition. At high concentrations the aroma was almost entirely lost. The content of reducing sugars, sucrose, non-sugar solids, vanilla resins, and the lead number were unchanged, and, except with very rapid distillation, there was no loss of vanillin. Anisyl alcohol and piperonal, absent from Mexican and Bourbon extracts, were present in Tahiti extracts both before and after distillation, and also in the distillate. Concentrates made from acetone extracts were inferior to those from alcohol due to the extraction of acrid matter. E. H. SHARPLES.

Studies in the genus *Mentha*. 12. Significance of the presence of γ-methyl-*n*-butyl alcohol in *Mentha piperita*, L., and its identification. S. M. GORDON (J. Amer. Pharm. Assoc., 1927, **16**, 130—135).—From an exhaustive examination of 150 lb. of "first runnings" from the distillation of peppermint oil it is concluded that *iso*butylcarbinol is present in American peppermint oil, and that it is formed by simultaneous reduction and oxidation of *iso*valeraldehyde, together with *iso*valeric acid. E. H. SHARPLES.

PATENTS.

Production of alcohols from methane. G. L. E. PATART (F.P. 593,650, 7.2.25).—Methane mixed with half its volume of oxygen is passed over coke or other combustible fuel at 800—1000°. The gas mixture is preheated to avoid any condensation or any fluctuations in temperature. The gaseous product, consisting of approximately 2 vols. of hydrogen to 1 vol. of carbon monoxide, is then converted into alcohols by the action of suitable catalysts at high temperatures and under pressure. Methane formed as a by-product is separated and utilised in the process. A. B. MANNING.

Catalytic production of methyl alcohol and liquid hydrocarbons. G. L. E. PATART (F.P. 594,121, 23.2.25).—A mixture of equal volumes of illuminating gas and water-gas is passed over a catalyst, consisting, *e.g.*, of zinc oxide and chromium oxide, at 300° and under 150—250 atm. pressure. On cooling, under the same pressure, a liquid product is formed which separates into two layers, the upper

containing hydrocarbons and the lower methyl alcohol. The gaseous product is returned to the initial reaction mixture. In the last hour of the reaction the amount of methyl alcohol produced is scarcely diminished, the pressure having fallen meanwhile to only 34 atm. It is better, therefore, to work at very high pressures (800—1000 atm.). Methane produced in the reaction is separated by refrigeration under pressure and submitted to the process of F.P. 593,650 (cf. preceding abstract). The residual gas mixture consists of hydrogen, nitrogen, and carbon monoxide. It is therefore possible to carry out the process without water-gas, using coal or coke-oven gas only. The liquid product may be used directly for heating or carburetting purposes, or the hydrocarbons may be separated after addition of water, and pure methyl alcohol obtained from the aqueous solution by distillation. A. B. Manning.

Synthesis of the higher alcohols. G. L. E. Patart (F.P. 593,649, 7.2.25).—The hydrocarbon product from the process of F.P. 594,121 (cf. preceding abstract) is passed over the same or another catalyst, the gases and reaction chambers being heated to avoid any condensation or undue temperature fluctuations. The higher alcohols are thereby produced in greater quantity than methyl alcohol. A. B. Manning.

Simultaneous production of methyl alcohol and liquid hydrocarbons. G. L. E. Patart (F.P. 593,648, 7.2.25; cf. preceding abstracts).—Gas mixtures containing hydrogen, carbon monoxide, and ethylene or its higher gaseous homologues, are submitted to the action of the catalysts used in the synthesis of methyl alcohol. Thus a mixture of 23% C_2H_4, 22% CO, 49% H_2, 1·2% CO_2, 0·5% O_2, and 4% N_2 was passed over a basic zinc chromate catalyst at 300° under 150—250 atm. pressure; the cooled reaction product formed two layers, of which the upper (about $\frac{1}{4}$) consisted of hydrocarbons of d^{15} 0·735, and possessed the fluorescence characteristic of some petroleum hydrocarbons. The lower layer was almost pure methyl alcohol, d^{15} 0·805, b.p. 66—68°. The process can be applied to gases produced by the cracking of petroleum or other oils. A. B. Manning.

Synthesis of the higher aliphatic alcohols, and their separation. G. L. E. Patart (F.P. 598,447, 9.4.25; cf. preceding abstracts).—Catalysts for the reduction of carbon monoxide or carbon dioxide by hydrogen under pressure at higher temperatures consist of an intimate mixture of alkali or alkaline-earth oxides with other metallic oxides. The former are obtained from the chromates, manganates, molybdates, tungstates, uranates, or vanadates of potassium, sodium, rubidium, barium, etc.; the number of atoms of alkali metal in the catalyst should amount to at least one half, or of alkaline-earth metals one quarter, of the total number of metallic atoms present. *E.g.*, zinc oxide is mixed with potassium chromate or manganese dioxide with neutral barium tungstate, the mixture made into a paste with 10% of a 10—20% dextrin or tragacanth solution, granulated, and dried at a temperature not higher than 320°; it is freed from dust before use. A suitable gas mixture contains about 50 volumes of hydrogen to 40 volumes of carbon monoxide. During the reduction, which is carried out at 330—400°, the carbon dioxide and methane formed are removed by refrigeration under high pressure. The liquid reaction product separates into two layers, an upper layer (about 30%) of d 0·885—0·890 and a lower aqueous layer of d 0·925—0·940. The alcohols in the lower layer are salted out at —20°, added to the upper layer, and the whole dried and distilled. The main fraction (about 60%) consists of propyl, butyl, and amyl alcohols; hexyl, heptyl, and higher alcohols are also formed. The salt solution contains methyl and ethyl alcohols. The products may be used as a source of the corresponding saturated hydrocarbons, aldehydes, etc., or may be used directly as liquid fuels.
A. B. Manning.

Preparation of acetone from acetic acid. Chem. Fabr. von. Heyden A.-G., and R. Feibelmann (G.P. 435,347, 12.2.22).—Acetic acid vapour is passed over cast-iron turnings in an iron tube at 400°, giving acetone and carbon dioxide. The acetone should be removed quickly from the tube by means of a slight suction. The coarseness of the catalyst facilitates the passage of the vapours, and so minimises the decomposition of the acid into methane and carbon dioxide. A good yield of acetone is claimed. C. Hollins.

Manufacture of *o*-aminoarylpropionic acid, its substitution products or homologues. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, und Brüning (E.P. 250,250, 31.3.26. Conv., 6.4.25).—Salts of *o*-aminoarylpropionic acids are obtained by hydrolysis of dihydrocarbostyril or its substitution derivatives with hot alkali. Thus dihydrocarbostyril itself, when heated for 12 hrs. at 150° with barium hydroxide and water, gives barium *o*-aminophenylpropionate, which may be converted into the sodium salt.
C. Hollins.

Manufacture of halogenohydrins. C. L. Long, F. G. Willson, and T. S. Wheeler (E.P. 265,259, 2.10.25).—The production of chlorohydrins from olefines and chlorinated water is not affected by concentrations of hydrochloric acid below which the formation of hypochlorous acid is depressed in accordance with the equilibrium $Cl_2 + H_2O \rightleftharpoons HCl + HOCl$. Halogenohydrins are produced by circulating water through a number of devices in some of which it is treated with a halogen, whilst in the others (alternately placed) the halogenated liquid is treated with an olefine at 0—20°. These steps are repeated until the concentration of halogenohydrin in the liquid reaches a value above which the formation of hypochlorous acid is depressed by the accumulated hydrochloric acid. This concentration (of chlorohydrin) should not in general much exceed a normal solution, and is continuously maintained by addition of water and withdrawal of treated liquid. Anhydrous halogenohydrins are obtained from the aqueous solution by conversion of the latter into the hydrocarbon oxide by known means, and treatment with dry hydrogen halide.
B. Fullman.

Preparation of halogenated alcohols. F. Nord (G.P. 434,728, 29.4.24; cf. Meerwein and others, A., 1925, i, 1239).—Halogenated aldehydes or ketones are reduced by aluminium alkoxides in the presence of aldehydes, such as acetaldehyde and benzaldehyde, in

non-aqueous diluents. Thus an equimolecular mixture of chloral and acetaldehyde, treated in absolute ether with aluminium *iso*amyloxide, gives 72—87% yield of trichloroethyl alcohol; bromal and benzaldehyde give 92% of tribromoethyl alcohol; *as*-dichloroacetone and acetaldehyde give 45% of dichloro*iso*propyl alcohol. C. HOLLINS.

Production of glycol monoethers. I. G. FARBENIND. A.-G., Assees. of BADISCHE ANILIN- U. SODA-FABR. (F.P. 610,282, 30.1.26. Conv., 24.4.25).—Olefine oxides are treated with alcohols or phenols in the presence of a catalyst, *e.g.*, sulphuric acid. Thus by passing ethylene oxide slowly into methyl alcohol containing 1% of sulphuric acid at 15°, glycol monomethyl ether results in 90% yield. The monoethyl ether is similarly prepared. C. HOLLINS.

Production of amide acid sulphates from nitriles. ROESSLER & HASSLACHER CHEMICAL CO. (E.P. 255,886, 22.7.26. Conv., 22.7.25).—Nitriles the radical of which is not affected by the acid used, are partially hydrolysed by dihydrated sulphuric acid (H_2SO_4,H_2O) to the amide acid sulphates. The reaction may be conducted in an inert liquid such as carbon tetrachloride, and is accelerated by the addition of small amounts of organic or inorganic halides such as sodium chloride, aniline hydrochloride, etc. A. DAVIDSON.

Production of esters from amide acid sulphates. ROESSLER & HASSLACHER CHEMICAL CO. (E.P. 255,887, 22.7.26. Conv., 22.7.25).—The sulphates of organic acid amides (cf. preceding abstract) react readily with alcohols (but not phenols) to form esters thus:— $R\cdot CO\cdot NH_2,H_2SO_4+R^1\cdot OH = R\cdot CO_2R^1+NH_4HSO_4$. The preparation of ethyl and allyl formates, benzyl acetate, propyl benzoate, glycol mono- and di-formates, and glyceryl monoformate is described. A. DAVIDSON.

Preparation of esters. I. G. FARBENIND. A.-G., Assrs. of M. MÜLLER-CUNRADI (G.P. 434,400, 15.2.24).—An alcohol, together with the ester of a second alcohol, is passed in vapour form over a heated catalyst, *e.g.*, alumina. Thus methyl formate and *iso*butyl alcohol, led over alumina at 150°, give *iso*butyl formate, b.p. 98°, and methyl alcohol, the conversion proceeding to the extent of 20—30%. From methyl formate and *iso*propyl alcohol at 200°, 10% of *iso*propyl formate is obtained; whilst amyl acetate and methyl alcohol, passed over precipitated silicic acid at 250°, give 10—15% of methyl acetate. C. HOLLINS.

Solutions of polymerised formaldehyde, free from paraformaldehyde, in mono- or poly-hydric alcohols. CHEM. FABR. VON HEYDEN A.-G., Assrs. of R. GEBAUER (G.P. 434,830, 6.5.23).—Solutions of paraformaldehyde or trioxymethylene in mono- or poly-hydric alcohols are heated with zinc oxide, alumina, or aluminium hydroxide, giving a *polymeride* of formaldehyde, m.p. 98°. The product is obtained in tallowy form on cooling, but liquefies at about 60°, and is then miscible with most organic solvents. The solutions may be advantageously used for the production of formaldehyde-phenol resins. The highly concentrated paste finds application as a desiccating agent for animal or vegetable preparations and for the removal of water by dialysis from water-sensitive colloidal solutions. The paste made with a suitable alcohol and an ointment base is absorbed by the skin with extraordinary rapidity. C. HOLLINS.

Production of sulpho-aromatic fatty acids. G. PETROFF (E.P. 252,212, 18.5.26. Conv., 18.5.25. Cf. E.P. 9160 of 1913; B., 1914, 652).—In the preparation of sulphoarylated fatty acids for fat-splitting etc., sulphuric acid and aromatic sulphonic acids are removed from the product of sulphonation of a mixture of aromatic hydrocarbons or phenols or turpentine with unsaturated fatty acids by washing with water and sodium chloride solution etc. The residue is dissolved in benzene, carbon tetrachloride, petroleum, etc., and the sulpho-aryl-fatty acid extracted with aqueous methyl or ethyl alcohol or acetic or formic acid. *E.g.*, a mixture of oleic acid, naphthalene, and benzene is treated with concentrated sulphuric acid, the reaction product mixed with water, and the upper layer, after washing with sodium chloride or sulphate solution, dissolved in a mixture of petroleum and benzene. The solution is washed with 15% sodium sulphate and 5% acetic acid, treated with 10% ethyl alcohol, and kept for 8—10 hrs. at 50—60°. The alcoholic layer, after washing with light petroleum and evaporation, yields the pure sulpho-aryl-fatty acid. B. FULLMAN.

Heavy metal salts of mercaptosulphonic acids. W. CARPMAEL. FROM CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING) (E.P. 266,824, 5.12.25).—Mercaptosulphonic acids or their soluble salts are treated in aqueous solution with a soluble heavy metal salt such as potassium bromoaurate, mercuric chloride, silver nitrate, bismuth chloride, etc. Examples are: thiophenol-*p*-sulphonic acid (from sulphanilic acid), 3-amino-thiophenol-6-sulphonic acid (from *m*-nitroaniline-6-sulphonic acid) and its formaldehyde-bisulphite compound, 1-mercapto-naphthalene-4-sulphonic acid, β-hydroxy-γ-sulphato-propyl mercaptan (from γ-chloro-β-hydroxypropyl hydrogen sulphate and sodium hydrogen sulphide), mercaptomethanedisulphonic acid, and 2-mercaptobenzoxazole-5-sulphonic acid (from *o*-aminophenol-*p*-sulphonic acid and potassium thiocyanate). The heavy metal salts of acylated aminomercaptosulphonic acids (E.P. 234,806; B. 1925, 692) are disclaimed. C. HOLLINS.

Preparation of salts of chlorourethane. W. TRAUBE (G.P. 435,529, 8.6.22).—N-*Chlorourethane*, $NHCl\cdot CO_2Et$, b.p. 101—102°/30 mm., prepared by chlorination of urethane in aqueous solution at 15°, is cautiously dissolved in *N*-alkali hydroxide. Evaporation yields the alkali salt; addition of silver nitrate solution precipitates the silver salt. *N*-Chloro-*N*-alkylurethanes are obtained easily by the action of alkylating agents on solutions of the alkali salts, *e.g.*, methyl sulphate yields N-*chloro*-N-*methylurethane*. The alkali salts of *N*-chlorourethane have bactericidal, fungicidal, and insecticidal properties. C. HOLLINS.

Manufacture of quinolinic [pyridine-2 : 3-dicarboxylic] anhydride. SOC. OF CHEM. IND. IN BASLE (E.P. 249,497, 2.3.26. Conv., 19.3.25).—Pure pyridine-2 : 3-dicarboxylic anhydride, m.p. 144—145°, in almost quantitative yield, free from pyridine-3-carboxylic acid, is prepared by heating the acid with acetic anhydride below 100°. Small additions of pyridine, hydrochloric

or sulphuric acid, acetyl chloride, a phosphorus halide, or an acid salt catalyse the reaction and lower the temperature necessary. B. Fullman.

Preparation of zinc compounds of aromatic biguanides. I. G. Farbenind. A.-G., Assees. of K. Schranz (G.P. 435,668, 11.9.24).—Arylbiguanides react with zinc oxide or hydroxide at the ordinary temperature, the former in the presence of water or alcohol, the latter by mere mixing, forming zinc salts which are vulcanisation accelerators. Examples are the *zinc* salts of *α-phenyl-*, o- and p-*tolyl-biguanides*, m.p. 195—196°, 182—183°, and 196—197°, respectively. C. Hollins.

Production of pure mercury vapour for inhalation purposes [from cinnabar, mercuric sulphide, etc.]. R. Spuhl (G.P. 435,689, 12.10.24).—The mercury salts are mixed with substances which, in presence of air, combine with the sulphur without the formation of volatile sulphur oxides. *E.g.*, 1 pt. of mercuric sulphide is mixed with 4 pts. of magnesium oxide, 2 pts. of sodium silicate, and 3 pts. of activated iron and the mixture formed into tablets. On heating these, pure mercury vapour is produced. The iron accelerates the reaction. E. H. Sharples.

[Magnesium] salt of a quinolinecarboxylic acid. M. L. Crossley, Assr. to Calco Chemical Co. (U.S.P. 1,618,172, 22.2.27. Appl., 20.9.24).—*Magnesium 2-phenylquinoline-4-carboxylate* is of therapeutic value in the treatment of rheumatism. T. S. Wheeler.

Manufacture of organic auro-mercapto-acids and salts thereof. W. Carpmael. From Chem. Fabr. auf Aktien (vorm. E. Schering) (E.P. 265,777, 15.2.26).—Auro-mercapto-acids and their salts are prepared by the action of auric salts on mercapto-acids or their salts in the presence of sulphurous acid or a sulphite. Using 1 mol. of sulphur dioxide per mol. of auric salt the mercapto-compounds are completely used up in the formation of auromercapto-compound, no by-product being formed. Examples are given of the preparation of sodium γ-auromercaptoglycerolsulphonate from the mercapto-compound and potassium bromoaurate and sulphurous acid or sodium hydrogen sulphite; and of the preparation of sodium *p*-auromercaptobenzenesulphonate and 4-amino-2-auromercaptobenzoic acid. B. Fullman.

Manufacture of basic bismuth salts of arylarsinic acids. R. W. E. Stickings, and May & Baker, Ltd. (E.P. 266,820, 5.12.25).—For the precipitation of basic bismuth salts in good yield and in a form suitable for purification, an alkali bismuthyl tartrate is heated with considerable excess of an arylarsinate in aqueous solution. The precipitate should be filtered off without delay in order to prevent gel formation. The products combine the therapeutic properties of bismuth and arsenic compounds, and have low toxicity. C. Hollins.

Production of concentrated acetic acid from dilute acid. H. Suida (U.S.P. 1,621,441, 15.3.27. Appl., 27.2.25. Conv., 8.3.24).—See E.P. 230,447; B., 1925, 827.

Manufacture of organic lead compounds. T. Midgley, jun., Assr. to General Motors Corp. (U.S.P. 1,622,228, 22.3.27. Appl., 19.5.23).—See E.P. 216,083; B., 1925, 299.

Manufacture of barbituric acid. F. Boedecker. Assr. to J. D. Riedel A.-G. (U.S.P. 1,622,129, 22.3.27, Appl., 23.9.24. Conv., 10.10.23).—See E.P. 223,221; B., 1925, 970.

Citric acid (G.P. 434,729 and E.P. 266,414—5).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Turbidimetry and grain size. F. F. Renwick (Phot. J., 1927, **51**, 185—193).—The properties of turbid media are considered in general, and a turbidimeter is described which determines the thickness of a medium which will just obscure a given test object. The instrument consists of a graticule immersed diagonally across a cell filled with the turbid liquid to be examined, and illuminated from the rear by diffuse light. The position of the last clear line of the graticule that can be resolved is measured and is used as a basis for calculating the obscuring thickness. Using this instrument, it is shown that the thickness of the layer of a turbid liquid which will just obscure the test pattern is inversely proportional to its concentration, or directly proportional to the volume containing unit mass of the suspended material. In the case of a silver bromide emulsion, the slope of the line connecting obscuring thickness with the volume of suspension containing unit mass of silver bromide varies over a wide range with change of size of the emulsion grains. Slow emulsions of intermediate grain size have the maximum covering power. The specific turbidity of a suspension is defined as the maximum area of a given contrast-pattern which can just be obscured by 1 g. of the suspension. If two emulsions of differing average grain characteristics are compared in the manner described, they differ considerably in their specific turbidities. Suspensions adjusted in dilution to have equal obscuring thicknesses have the same obscuring thickness after admixture, no matter how widely they differ in specific turbidity, or in what proportions they are mixed. Formulæ are given for determining the obscuring thickness of mixtures with known individual values, and for the calculation of the specific turbidity of a mixture of two different grain types, if the connexion between specific turbidity and grain size were known. There is a marked difference between the obscuring power of a gelatin–silver bromide layer in the wet and dry state, and the ratio of their turbidities varies greatly with grain size. The descending order of turbidity ratio does not strictly correspond with that of diminishing speed, and it is not certain whether it corresponds with their average grain sizes. A second turbidimeter is described, the principle of which depends on the use of a comparison wedge of diffusing medium such as a gelatin wedge of bismuth oxychloride or silver iodide. W. Clark.

Patents.

Production of photographic plates, films, and the like for indirect three-colour photography,

E. A. LAGE (E.P. 266,468, 28.11.25).—A film, coated with emulsion suitable for the red partial image, is squeegeed on to a support provided with a coating of emulsion suitable for the blue partial image, the emulsion surfaces being in contact, after which a coating of emulsion suitable for the yellow partial image is applied. After exposure, the film, together with the final coating, is stripped, and the two parts are developed separately, and dried. One or both negatives are then stripped from the film. In this way three partial negatives are obtained for printing. Alternatively, one or other of the partial images on the film which has been separated from the support is rendered translucent by bleaching, the unbleached negative is printed, and the bleached image is then redeveloped. W. CLARK.

Photographic prints in colours. G. P. J. SCHWEITZER (E.P. 249,530, 17.3.26. Conv., 18.3.25).—Coloured photographic images are produced by dyeing a plate carrying a silver image, and then attacking the silver with solutions capable of freeing, in this attack, a reagent which bleaches the dye by oxidation. A solution containing sodium bromate and sulphuric acid is suitable. Alternatively, a solution capable of reacting with silver halide to free a reagent which bleaches the dye is used. Such a solution contains iodic acid, sulphuric acid, and iodine, and it is used on a developed but unfixed plate. The dye is bleached according to the amount of silver or silver halide present. W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Propagation of detonation across an air-gap between two cartridges of explosive. G. ST. J. PERROTT and D. B. GAWTHROP (J. Franklin Inst., 1927, **203**, 387—406).—Experiments with a Mettegang recorder show for explosives detonating at about 1700 m./sec. that the velocity of propagation across an air-gap between two cartridges is at first much higher than the rate of detonation of the explosive and falls off regularly until the second cartridge fails to detonate. When the rate is about 3000 m./sec. the velocity across the air-gap is about the same over an interval of $4\frac{1}{2}$—$7\frac{1}{2}$ in. from the end of the initiating cartridge. With a rate of detonation as high as 4800 m./sec. the velocity in the air-gap falls off linearly as the gap is increased from $1\frac{1}{2}$ to 27 in. The decrease is slow from 27 to 48 in., and the second cartridge will not always detonate at 48 in. Explosives containing ethylene glycol dinitrate propagate across a greater air-gap than do similar explosives containing nitropolyglycerin, and maintain a higher average velocity across the air-gap. Photographs on a moving film confirm the above results. When the explosives are confined in glass tubes the speed of the disturbance is considerably greater than the rate of detonation of the explosive, and is maintained for a longer distance. A luminous phenomenon is propagated through the air-gap, and is in some way related to the disturbance effecting detonation in the second cartridge. R. A. MORTON.

PATENT.

Explosive. A. SEGAY (U.S.P. 1,620,696, 15.3.27. Appl., 30.4.23. Conv., 26.5.22).—See E.P. 201,791; B., 1923, 999 A.

XXIII.—SANITATION; WATER PURIFICATION.

Hufschmidt's process for regenerating permutit filters. K. HOFER (Glückauf, 1926, **62**, 1396—1398; Chem. Zentr., 1926, II, 2993).—The addition to an existing permutit plant of rinsing tubes and a pump for circulating the regenerating solution resulted in a considerable saving of sodium chloride and decreased the time of regeneration from 9 hrs. to $\frac{1}{2}$ hr., whilst the addition of a gravel filter and a froth trap reduced the loss of permutit to practically nil. L. A. COLES.

PATENTS.

Apparatus for preparing lime water for use in water-purifying plants. H. MORGENSTERN and W. HAGEN (G.P. 435,722, 13.1.25).—Water is added to lime suspended at the bottom of a funnel-shaped vessel, through a pipe which passes downwards through the liquor and then bends round and is connected with a length of wider upright tube, a suction pipe dipping into the suspension being attached at the point of connexion of the two tubes. L. A. COLES.

Filtration process. H. W. BLAISDELL, Assr. to BLAISDELL FILTRATION Co. (U.S.P. 1,614,947, 18.1.27. Appl., 12.12.21).—At intervals between the cleanings of a filter, a reverse flow is allowed at a low rate insufficient to remove sediment on the bed, but sufficient to cause relative re-arrangement of the material and sediment. W. T. LOCKETT.

Reconditioning zeolite water softeners. W. J. KENNEY, Assr. to ZEOLITE ENGINEERING Co. (U.S.P. 1,617,288, 8.2.27. Appl., 16.3.25).—To facilitate the reconditioning of zeolite water softeners means are provided to cause a portion of the liquid discharged from the treating reservoir during regeneration to be passed through a salt pot and recirculated through the reservoir without placing the interior of the salt pot either under the pressure of the dispensing system or that of the water supply main. W. T. LOCKETT.

Improving the odour of phenols and hydrocarbons used for disinfectants. ZECHE M. STINNES (G.P. 435,692, 23.10.24).—To the phenols or hydrocarbons, with or without the addition of solutions of soap or other emulsifying agents, are added ethers or esters in sufficient amount to mask the odour of the disinfectant, the bactericidal power of which is thereby unaffected. E. H. SHARPLES.

Germicidal agent. I. G. FARBENIND A.-G., Assees. of M. MÜLLER-CUNRADI and K. VIERLING (G.P. 435,755, 4.3.24).—A mixture of alcohol and a butyl alcohol such as, *e.g.*, *n*- and *iso*-butyl alcohol, suitably diluted with water, is used. E. H. SHARPLES.

Disposal of sewage. C. P. DERLETH, Assr. to CELITE Co. (U.S.P. 1,617,014, 8.2.27. Appl., 7.2.22.—In the activated sludge sewage disposal processes to facilitate the purification of the sewage, the settlement of the sludge in the settlement tanks, and the filtration of the surplus sludge in filter presses and the like, a quantity of diatomaceous earth is added to the sewage or the mixed liquor prior to aeration. W. T. LOCKETT.

Disinfectant composition. G. G. GORDON (U.S.P. 1,619,861, 8.3.27, Appl., 17.12.26. Conv., 29.6.25).—See E.P. 258,678; B., 1926, 998.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 27, 1927.

I.—GENERAL; PLANT; MACHINERY.

Heat insulation. J. S. F. Gard (J.S.C.I., 1927, 46, 101—105 T).—The qualities required of a heat insulation are low heat conductivity, low specific heat, low sp. gr. to avoid strain on pipes etc., mechanical strength, non-inflammability, absence of corrosive action on metals, ability to withstand alternate wetting and drying and heating and cooling without cracking. The materials giving best all-round results are: (1) For refrigeration etc., sheet and pipe sectional cork moulded from virgin cork without artificial binders. The product is waterproof, resists the action of organic solvents, can be worked with joiners' tools, is resistant to fire, and is rot- and vermin-proof. (2) For steam pipes, boilers, etc. at temperatures up to 700° F., 85% magnesia covering (15% of asbestos fibre and 85% of basic magnesium carbonate). (3) For superheated steam plant etc. at 700—1000° F., a modified form of the above, consisting of two layers—one of a special form of magnesia lagging, resistant to higher temperatures, of ½ in. thickness, which breaks down the temperature to 700° F.; the second a layer of 85% magnesia to the required thickness. (4) For furnace work, a honeycombed brick, consisting chiefly of kieselguhr. All these materials give a relative efficiency of over 90% against an uncovered metal surface. Methods of testing are described and examples of savings effected are worked out.

Direct determination of distribution curves of particle size in suspensions. E. M. Crowther (J.S.C.I., 1927, 46, 105—107 T).—During the process of sedimentation from a dilute suspension, the concentration or density at a given depth x after a time t is a measure of the amount of particles which have settling velocities equal to or less than x/t. Measurements of the density of the suspension at a given depth as a function of time can provide directly a continuous size-distribution curve of sedimenting particles. The difference of hydrostatic pressure over a small range near the base of a column of suspension may be taken as the mean density over this range. An apparatus is described for obtaining such pressure differences by direct readings on a highly sensitive differential liquid manometer, consisting of aniline in contact with slightly alkaline water. The difference of the aniline levels in the manometer gives the proportion of material remaining in suspension at a known depth and time, and having therefore particle sizes equal to or less than a known value. Summation percentage curves are readily constructed by plotting these readings against the corresponding times or their logarithms. A simple change of scale gives the settling velocities and, in the case of the logarithmic curve, the logarithm of the equivalent diameters, as deduced by Stokes' law.

Active silicic acid. Ruff and Mautner.—See VII.

Patents.

[Refractory heat screen for] furnaces. Carborundum Co., Ltd. From Carborundum Co. (E.P. 266,140, 4.3.26).—In a furnace which has "radiating combustion chambers" separate from that containing the goods, the refractory walls between the combustion and goods chambers are constructed in zones having different thermal conductivities, so that a gradient in the heat transfer to different portions of the furnace may be obtained. B. M. Venables.

Apparatus [tunnel kiln] for burning or calcining materials. H. M. Robertson (U.S.P. 1,621,222, 15.3.27. Appl., 20.6.25).—In a tunnel kiln which is provided with a preheating section and a longer burning section, the latter is heated by flames from primary combustion chambers built in the side walls, which flames enter the lower part of secondary combustion chambers, mix with more air, and emerge through ports extending the whole length of the burning section. B. M. Venables.

Drying apparatus. G. W. Christoph, Assr. to American Hardware Corp. and Sterling Blower Co. (U.S.P. 1,623,017, 29.3.27. Appl., 14.12.25).—The goods to be dried are tumbled in drying material within a drum, the drying material is removed by a concentric screen, and returned by another concentric drum to the point of entry. B. M. Venables.

Treatment of hygroscopic materials. E. C. R. Marks. From Carrier Engineering Corp. (E.P. 267,215, 9.12.25).—Material which it is desired to deliver with a definite proportion of moisture is conveyed through an apparatus of three compartments, the atmosphere in each compartment being controlled by air leaving that compartment. In the first compartment the temperature is controlled by a thermostat, but the humidity is not regulated. In the second damping sprays and heating means are provided, and the humidity is regulated by wet and dry bulb thermometers. The air going to the third compartment is previously saturated and then heated (under control of a thermostat) just before entering the chamber. B. M. Venables.

Heat treatment of materials. [Ore reduction or coal distillation.] J. Prentice and A. P. Pehrson (E.P. 266,402, 16.4.26).—The material constitutes a heating resistance between metallic or carbonaceous plate electrodes within a rotary or oscillatory furnace

chamber provided at its opposite ends with inlet and outlet necks for maintaining a uniform level of the material in the chamber. In treating material which becomes sufficiently conductive only when heated, a resistor material, *e.g.*, coke, carborundum, or calcined anthracite, may be preliminarily supplied to the furnace until the desired temperature is reached. The material is preferably fed continuously by a screw conveyor through a preheating chamber to the inlet neck of the furnace chamber, the residues and volatile products being continuously withdrawn and cooled. The bore of the furnace chamber between the electrodes may be made of varying cross-sectional area to obtain local variations of temperature. H. HOLMES.

Ball or tube mill. C. VON GRUEBER MASCHINENBAU A.-G., and C. PFEIFFER (E.P. 262,785, 8.12.26. Conv., 8.12.25).—A ball or tube mill is divided into two grinding compartments by a transverse division comprising two walls and a narrow space between. The material from the first grinding chamber passes through concentric ring-shaped slots in one wall into the space, where it is lifted by curved vanes to a central opening in the other wall, through which it passes into the second grinding chamber. A central opening may be made also into the first chamber, and within these openings may be placed a double cone deflector which, in conjunction with the slope of the vanes, is stated to have a certain classifying effect, returning large material to the first grinding chamber. B. M. VENABLES.

Grinding or pulverising mill. GEBR. SCHLEIFENBAUM & Co., G.M.B.H., and A. IRMER (E.P. 267,426, 27.10.26).—A disintegrator which is specially suitable for coal and other fuel is formed with the stationary impact surface of round rods in loose contact with each other and free to revolve under the impact of the material. A number of disintegrating chambers may be assembled in line, each one having beaters of a slightly larger diameter than the preceding (coarser) one. B. M. VENABLES.

Comminuting mill. R. C. NEWHOUSE, Assr. to ALLIS-CHALMERS MANUF. Co. (U.S.P. 1,620,982, 15.3.27. Appl., 15.4.26).—A grinding mill, of the ball or pebble type, is divided into two compartments, viz., a cylindrical primary reduction chamber of larger diameter (containing larger balls) tapering to a secondary cylindrical portion of smaller diameter, with a perforated partition between the two. B. M. VENABLES.

[Screen] separator. T. J. STURTEVANT, Assr. to STURTEVANT MILL Co. (U.S.P. 1,621,564, 22.3.27. Appl., 29.6.25).—A substantially flat screen cloth rests without being stretched upon a frame with round cross bars which is vibrated. B. M. VENABLES.

Filters [for sewage and industrial effluents]. A. C. HANDLEY (E.P. 266,857, 6.1.26).—An open tank is provided with removable filter elements extending transversely or transversely and longitudinally across it and comprising coir fibre between expanded metal sheets, which may be connected by spacing bolts. H. HOLMES.

Edge filters. AKTIEBOLAGET SEPARATOR (E.P. 244,111, 4.12.25. Conv., 4.12.24).—Laminæ for edge filters are made of thin sheets of metal which have been locally reduced in thickness by corrosion or erosion. The sheets are assembled with the unreduced portions in line. B. M. VENABLES.

[Fritted glass] filter. SCHOTT & GEN., Assees. of M. HERSCHKOWITSCH and P. PRAUSNITZ (U.S.P. 1,620,815, 15.3.27. Appl., 12.6.24. Conv., 27.6.26. Cf. E.P. 218,286 and 226,182; B., 1925, 230; 1926, 423).—A filter comprises a porous plate of fritted glass fused into a funnel made of glass having the same coefficient of thermal expansion. T. S. WHEELER.

Distilling apparatus [bubbling column]. W. E. BROWN, Assr. to UNITED IRON WORKS, INC. (U.S.P. 1,620,593, 8.3.27. Appl., 6.8.24).—The gases pass upwards through a casing which is provided with a number of liquid holders or trays, each comprising a number of parallel troughs, through which the liquid passes zig-zag horizontally in each tray, then zig-zag downwards from tray to tray. The openings between the troughs are covered by hoods, which cause the gases to bubble through the liquid. B. M. VENABLES.

Evaporator. C. W. AIKEN (U.S.P. 1,621,862, 22.3.27. Appl., 5.4.23).—A quantity of liquid contained in a casing is heated by a steam drum or nest of vertical tubes in the lower part, the steam being admitted through the upper tube plate into a telescopic nozzle tube, which has discharge nozzles in the lower part of the drum. B. M. VENABLES.

Method of emptying containers. I. G. FARBENIND. A.-G., Assees. of FARBENFABR. VORM. F. BAYER & Co. (E.P. 249,475, 8.1.26. Conv., 23.3.25).—A container, as described in E.P. 247,744 (B., 1926, 345), is provided, immediately below the nozzle, with a constricted channel shaped as a solid of revolution and directed downwards towards one or more discharge passages. Preferably the bottom of the container is fitted with a discharge funnel having a cylindrical portion into which projects a conical member forming a bearing for the rotary pipe carrying the nozzle. Removal of coarse or moist material is facilitated. H. HOLMES.

Apparatus for the mechanical production of colloidal material. SUDENBURGER MASCHINENFABR. & EISENGIESSEREI A.-G., and W. OSTERMANN (G.P. 436,368, 28.3.25. Addn. to G.P. 421,318).—The outer casing and grinder inside it are both provided with grooves parallel to the axis, which decrease in depth towards the outlet, a short length at the end being ungrooved so that the material has to pass between the grinder and the casing. There are also one or more grooves cut at right angles to the axis. L. A. COLES.

Production of impervious [graphitic] material. W. F. BLEECKER (U.S.P. 1,620,940, 15.3.27. Appl., 28.12.21. Cf. U.S.P. 1,462,003; B., 1923, 832 A).—Artificial graphite is heated *in vacuo*, and molten asphalt, tar, or the like is then admitted so as to impregnate the graphite. The material is then baked out of contact with oxygen, and the process is repeated if necessary. The product is of value as a chemically-resistant material. T. S. WHEELER.

Method of heating furnaces. F. HELBIG (U.S.P. 1,625,082, 19.4.27. Appl., 21.3.23. Conv., 17.5.21).—See E.P. 211,548; B., 1924, 382.

Absorption refrigerating apparatus. B. C. von Platen and C. G. Munters, Assrs. to Electrolux Servel Corp. (U.S.P. 1,620,843, 15.3.27. Appl., 4.8.23. Conv., 18.8.22).—See E.P. 202,650; B., 1925, 975.

Comminuting mill. Allis-Chalmers Manuf. Co., Assees. of R. C. Newhouse (E.P. 268,264, 29.12.26. Conv., 15.4.26).—See U.S.P. 1,620,982; preceding.

Method and apparatus for grading solid materials. G. R. Baker, W. E. Prescott, and C. W. Gilderdale, Assrs. to Rowntree & Co., Ltd. (U.S.P. 1,623,040, 5.4.27. Appl., 31.1.24. Conv., 26.2.23).—See E.P. 220,671; B., 1924, 895.

Condensing apparatus. A. O. H. Petersen, Assr. to N. V. Nederlandsche Installatie Maatschappij Therma (U.S.P. 1,624,066, 12.4.27. Appl., 20.5.25. Conv., 17.1.24).—See E.P. 243,982; B., 1926, 113.

Hammers for grinding, crushing, and pulverising mills. Bossert Corp., Assees. of W. J. Clement (E.P. 260,269, 20.10.26. Conv., 20.10.25).

Cleaning high-pressure boiling apparatus. G. Ullmann (E.P. 257,600, 19.8.26. Conv., 28.8.25).

Producing foam for fire extinguishing purposes. Excelsior Feuerlöschgeräte A.-G., Assees. of Minimax A.-G. (E.P. 254,285, 1.6.26. Conv., 29.6.25).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Coke in relation to some of its industrial and domestic uses. H. Hollings and N. E. Siderfin (J.S.C.I., 1927, **46**, 76—84 t).—A rapid method for determining reactivities of coke is described, which is designed to reveal small differences of reactivity and the progressive changes which occur in any one coke during prolonged heating at 950° in contact with carbon dioxide. The gas is passed continuously at constant rate over the coke sample at 950°. The reactivity at any given time during the test is obtained by noting the velocity of the gas after its passage over the coke relative to its initial velocity, and calculating therefrom the change in volume which has occurred due to carbon monoxide formation. The reactivity is expressed as percentage of carbon dioxide converted. The cokes tested, arranged in order of their reactivities, were as follows:—Coke from blended coals; vertical-retort coke treated with sodium carbonate solution; horizontal-retort coke similarly treated; vertical-retort coke; horizontal-retort coke; beehive oven coke. The general properties of "hard" and "soft" cokes are discussed. Six different fuels were tested in closed stoves for combustibility in air, the basis for comparison being the minimum quantity of air required to maintain combustion during a 10-hr. run. Two stoves with grates 24 in. and 8 in. diameter, respectively, were used, and it was found that whereas "hard" coke was most suitable for the large stove, the "soft" cokes were preferable for the small stove because their higher combustibility promoted easier control. The control is rendered easy on the large stove, not by good combustibility, but by the insulating effect of the coke surrounding the central core of burning fuel. It is suggested that by carbonising a suitable blend of coals in suitable plant (*e.g.*, coke ovens) it should be possible to produce a hard coke which is also highly combustible and reactive. The efficiency of water-gas manufacture as influenced by quality of coke and air-blast velocities is discussed with reference to experimental data published in the Carbonisation Committee's Report to the American Gas Association. It is shown that there is less loss of heat in the blow gases when using a coke of low reactivity than when using a more reactive fuel, and that with any given fuel the efficiency of the blow is increased by increasing the velocity of the blast.

Coke formation. R. A. Mott (J.C.S.I., 1927, **46**, 85—92 t).—The results are recorded of an experimental study of coke formation from lump coal and crushed fine coal for a series of 17 representative British coals. The coals chosen cover a range of carbon contents of 92 to 79% for bright coals (clarains) and 88 to 81% for hard coals (durains). The standard for coking power chosen is the ability to produce in practice a metallurgical coke from crushed fine coal. It is shown that coals which produce a serviceable coke in practice have considerable swelling power when heated at fast or slow rates of heating, whilst inferior coking coals swell less considerably, and non-coking coals show no marked swelling power. It is argued that this swelling power (which is due to the evolution of volatile matter during the plastic stage of coke formation) is an important factor in conferring mechanical strength to the resultant coke in coke-oven practice. It is suggested that coke formation is due to vitrification when the molecules of the solid surfaces gain under mechanical disturbance (due to the swelling power) a temporary and local mobility approaching that of the liquid state. It is also suggested that in coke formation from crushed coal, wetting phenomena are important, and aggregation of the various particles into a strong coherent residue can only occur if liquid hydrocarbons extruded from the particles remove the air films from their surfaces. If swelling also occurs at this stage, the molecular force of cohesion can act and aggregate the particles into a coherent residue. In the formation of coke from lump coal (such as occurs in vertical-retort practice) it is argued that the opposing forces, swelling power and cohesion, determine the strength of the resultant coke.

Behaviour of carbonised fuels in the open fire-grate. (Mrs.) M. F. Bligh and H. J. Hodsman (J.S.C.I., 1927, **46**, 92—99 t).—The radiant efficiency was measured for several solid fuels burning in an open domestic grate adapted to consume carbonised fuels. The importance of heat-insulation of the fire-back was noted. The cokes usually radiated more of their heat of combustion than the coal. The size of coke had little effect on the efficiency, but large pieces were more difficult to ignite. The radiant efficiency of a low-temperature coke (9·5% volatile matter) was greater than that of the coal, but less than that of a gas coke (vertical retort). The highest figure (29%) was obtained with a special coke of very low ash content prepared under high-temperature conditions. A by-product coke was difficult to ignite, and it burned only so long as a deep column of fuel was maintained. It is considered

that if a high radiant efficiency is sought a low content of ash and of volatile matter should be aimed at. Impregnation of the gas coke with sodium carbonate increased its rate of combustion, but lowered the thermal output by radiation.

Heat of combustion of the carbon in coke, and reactivity of coke. M. W. TRAVERS (J.S.C.I., 1927, **46**, 128—130 T).—The results of calorific value determinations of samples of coke produced under known conditions are analysed. It is shown that the derived heat of formation of carbon dioxide is generally lower for high-temperature coke than for low-temperature coke. It is suggested that a practical limit to the value of this constant for coke produced in modern plant will be in the neighbourhood of 95,000 C.H.U. per lb.-mol. or 14,250 B.Th.U. per lb. of carbon. So long as the material is porous the surface energy will be considerable, and the value for massive graphite, 94,275, will not be reached.

Method of ignition in calorimetric determinations of calorific value. O. KÜNLE (Brennstoff-Chem., 1927, **8**, 107).—The powdered substance is enclosed in a wrapper made of cigarette paper, weighing about 0·05 g., and having a calorific value of about 155 kg.-cal. The packet is wound round the centre and at each end with the ignition wire, and is suspended between the ignition terminals in the calorimeter. In the case of substances which are difficult to ignite, a wrapper of waxed paper may be preferable (cf. Steuer; B., 1927, 161).
W. T. K. BRAUNHOLTZ.

Determination of nitrogen in fuels. G. LAMBRIS (Brennstoff-Chem., 1927, **8**, 108; cf. B., 1927, 289).—A correction of a printer's error in the original article.
W. T. K. BRAUNHOLTZ.

Thermal study of the manufacture of water-gas. A. PARKER (J.S.C.I., 1927, **46**, 72—76 T).—The principal thermal changes involved in the manufacture of blue water-gas by the intermittent process are discussed. A thermal account constructed from data procured during an investigation of the process is given. The total heat supplied to the generator in the form of coke, steam, and air is accounted for in eight items: (1) Potential heat value of the water-gas made; (2) sensible heat of water-gas; (3) total heat of undecomposed steam; (4) potential heat of blow gas; (5) sensible heat of blow gas; (6) potential heat of ashes, clinker, and dust; (7) sensible heat of ashes, clinker, and dust; (8) a difference item including losses by radiation etc. A suggested scheme for separate thermal analyses of the air-blow periods and steaming periods is discussed, and a new scheme, which does not require assumptions regarding the nature of coke carbon, is proposed. Examples of separate thermal accounts are given.

Study of flame movement. O. C. DE C. ELLIS (Safety in Mines Res. Brd., Paper No. 32, 1927, 35 pp.).—The methods of measuring flame speeds are discussed. An improved photographic method working on the cinematographic principle is described. A series of apertures on the periphery of a rapidly revolving disc passes before the lens of a plate camera. A similar, auxiliary disc revolves before this in the reverse direction, so that the gradual obscuring of the lens ends at its centre and not at its rim. A radial knife device, which can be released at will, is attached to the first disc. When in use, the two discs are revolved at a constant known speed, and the flashes from the mercury break of an electrically-controlled tuning fork are registered on the revolving drum of an auxiliary camera (which thus serves both as a chronograph and to record the speed of propagation of flame). When the radial knife is released, (i) the primary of an inductively-coupled circuit is ruptured and immediately re-made, thus causing a spark which is registered on the revolving drum; (ii) just afterwards, a second, similar circuit is broken, the resulting spark igniting the gas mixture; and (iii) the first circuit is again broken when the wheel has completed half a revolution, causing another flash to be registered on the drum. Thus the drum record of sparks and of the time scale gives the necessary information for calculating the time interval between ignition and the first exposure on the plate, and the intervals between successive exposures. A series of photographs on the same plate can be taken at accurately-timed intervals during the course of an explosion, or a series of explosions can be carried out under identical conditions and a number of photographs taken at chosen time intervals on separate plates. Both methods of application are illustrated by photographs of the flames in mixtures of air or oxygen with carbon monoxide.
S. K. TWEEDY.

Estonian shale oil. I. Isolation and properties of phenols. P. N. KOGERMAN (J.S.C.I., 1927, **46**, 138—143 T).—Estonian oil shale, "kukersite," on distillation at 500—600° affords about 20% of its weight of crude oil, d^{15} 1·007, viscosity (Engler) 5·5°, moisture 0·9%, containing 72·1% of neutral substances, chiefly hydrocarbons, 22·4% of phenols, 4·0% of carboxylic acids, and 0·2% of bases. Investigation of the fraction of b.p. 230—270°/760 mm. (the lowest containing phenols in quantity) indicates a tendency for the phenolic content to decrease on redistillation. This fraction, which forms 8% of the crude oil, contains 17·2% of phenols, 2·5% of acids, and less than 1% of bases. The neutral oil, yellow, has d^{20} 0·857 (d^{17} 0·847 on redistillation *in vacuo* at 60 mm.), and when treated with liquid sulphur dioxide at —20° affords 32·8% of refined oil, d^{17} 0·7995, $n_D^{20.5}$ 1·4471, 35% of which is absorbed by sulphuric acid; the extract (61·2%, has d^{17} 0·8663, $n_D^{20.5}$ 1·4792, and 70% is absorbed by sulphuric acid. The phenolic fraction contains *o*-, *m*-, and *p*-cresols, 1 : 4 : 5-, 1 : 2 : 4-, and 1 : 3 : 4-xylenols, and the presence of mesitol or propylphenol is indicated. Pyrocatechol is present in the aqueous fraction obtained on distillation of the oil. The phenols of kukersite thus resemble those of low-temperature coal tar (Weindel; B., 1925, 795), but, in addition, phenolic ethers are present. Guaiacol has been detected qualitatively. With strong sodium hydroxide solution the fraction, b.p. 230—270°, is completely resinified. For the extraction of phenols from the crude oil, the oil is mixed with an equal volume of benzene, and the concentration of the sodium hydroxide solution should not exceed 15%. The quantity of oil dissolved by the latter reagent increases both with the quantity and concentration of the sodium hydroxide solution. Variations were not so great on extracting the fraction of b.p. 230—270°. For the determination of the phenols,

the water content is determined by distillation with xylene and also the matter insoluble in ether; a second sample of equal weight is distilled up to 160°, the remaining oil dissolved in a measured volume of ether, and extracted three times with 10% sodium hydroxide solution. The extract is washed with an equal volume of ether, and the washings are added to the oil. The ether is evaporated *in vacuo*. The flask containing the oil is warmed for 10 min. and weighed, the water being determined by xylene distillation. R. Brightman.

Determination of paraffin wax in crude wax. L. M. Henderson and S. W. Ferris (Ind. Eng. Chem., 1927, **19**, 262—264).—Various methods for the analysis of oil-wax mixtures are reviewed. A modification of the method of Wyant and Marsh (B., 1926, 37), using nitrobenzene as a solvent, is proposed. The determination is carried out in a Kjeldahl flask with shortened neck, to the bottom of which a portion of the bottom of a test tube is fused. A surrounding beaker provides an air bath for hot or chilled air. The flask is fitted with a thermometer, a tube for the withdrawal of the dissolved oil, and a suction tube for vapours. 20 c.c. of nitrobenzene are added to a 10 g. sample in the flask, air is slowly bubbled through, and the temperature raised to 70°, and then lowered to 32° for 5 min. The wax solidifies and floats on the surface of the solvent. The oil solution is withdrawn, and a second 20 c.c. of solvent are added, and the temperature is raised to 135° and later to 150°. Nitrobenzene vapours are removed by suction, when the m.p. of the wax may be determined, and the yield of wax weighed. The yields closely agree with those of sweating ovens. H. Moore.

Specific gravity of paraffin wax. F. J. Morris and L. R. Adkins (Ind. Eng. Chem., 1927, **19**, 301—302). —The minute bubbles in commercial wax contain air, which, however, is not held in solution when wax is melted at atmospheric pressure. A modified form of the Nicholson hydrometer made of glass was used to determine sp. gr. The pan in the liquid consisted of two crystallising dishes one inside the other, the inner one, inverted, having two V notches in the edge to allow of the flow of water. Samples of wax were kept under 30 mm. pressure for 24 hrs. at 60—65°, and cooled *in vacuo* at room temperature. Samples were weighed on the instrument in air and in water at 15·5°. The results differed greatly from those taken at 54·5° and reduced to 15·5° by the petroleum conversion tables. To determine the expansion coefficient, sp. gr. were taken at every 5·5° from 15·5° to 43°, and at every 2·3° from 43° to 54·5°. The results, given in graph form, show the break in the curve where most of the hydrocarbons of the sample melt, and also flat spots of unknown significance. H. Moore.

Deterioration of mineral oils. I. Mechanism of oxidation and action of negative catalysts as determined by a dynamic method. R. T. Haslam and P. K. Frolich (Ind. Eng. Chem., 1927, **19**, 292—296). —Tests were made on a highly refined Parke-Davis oil. Dry oxygen was bubbled at 10 litres per hr. through 75 g. samples in large test tubes, the temperature being kept at 130° or 140°. The resulting acidity was titrated as oleic, and the viscosity taken by Lang's method. The action of catalysts was expressed by the hours by which oxidation was accelerated or retarded. The addition of 0·01 g. of a negative catalyst gave a delay in hours as follows for the substances tested: β-naphthylamine, 3; ethyl-α-naphthylamine, 3; methyl-α-naphthylamine, 4·5; α-naphthylamine, 4·5; *p*-aminophenol, 5; diphenylamine, 5·5; phenyl-α-naphthylamine, 11, and diphenylhydrazine, 18. The oxidation curve after the delay has the same form as with the pure oil. Experiments on the loss of power of the catalyst were made, using heat alone, oxygen, and nitrogen. *p*-Aminophenol disappears by evaporation; diphenylamine is mainly destroyed by heat, whilst diphenylhydrazine is partly evaporated and partly destroyed by oxidation. Preheating with nitrogen accelerated oxidation with oxygen. The rate of oxidation was the same with dry as with damp oxygen. Oxidation curves tend to attain a maximum at about 28 hrs., and rise only slightly up to 40 hrs. The viscosity curves show similarity with the oxidation curves, but the viscosity increases more rapidly, particularly after the oxidation curve has reached its maximum. Only a small proportion of the oxygen in oxidised oils is present as acids, the rest being probably as products of condensation or polymerisation. Aldehydes are not stable in the heated and oxidising oil. The process of oxidation appears to follow the series:—hydrocarbons, alcohols, aldehydes, ketones, naphthenic and fatty acids, condensation and polymerisation products. H. Moore.

Evaluation of turbine oils. T. H. Rogers and C. E. Miller (Ind. Eng. Chem., 1927, **19**, 308—312).— Deterioration of turbine oils in service is due to oxidation, with the formation of asphaltic material insoluble in oil, and of free acids soluble in oil, which form insoluble soaps in contact with iron or copper. Both the soaps and the asphaltenes are emulsifying agents of the water-in-oil type. Turbine deposits consist mainly of heavy-metal soaps. A proposed stability test consists in subjecting 500 c.c. of oil to oxygen (2 or 3 bubbles per sec.) at 100°. Acidity and Herschell demulsibility tests were taken at intervals of 48 hrs. The test is made in a flask supported in a water bath, and fitted with a thermometer and tube for oxygen supply. Acidities were determined by titration, using 95% alcohol and phenolphthalein as indicator. The standard tests were made with a spiral of iron wire present. The acidity curves do not always conform with the demulsibility curves. Confirmatory tests were run on 1000-kw. turbines over a period of some 7 months; 1 hr. of stability test was roughly equal to 9 hrs.' running. The stability tests were confirmed by the behaviour of the oil in the dry turbine. H. Moore.

Prediction of flash point of blends of lubricating oils. E. W. Thiele (Ind. Eng. Chem., 1927, **19**, 259—262).—An approximate method of calculation is based on the principle that the antilogarithm of one-hundredth part of the flash point is an additive property of the oil (on a volume basis), the flash point of an ordinary mixture of lubricating oils being calculated from the values of its constituents. Tables are given for mixtures of two oils. Assuming that the flash point is the temperature at which the vapour pressure of an oil reached about 10 mm. of mercury, the author demonstrates

mathematically the correctness of the method, which does not, however, apply to oils of low b.p., *e.g.*, kerosene. H. MOORE.

Acids in automobile crank-cases. A. F. MESTON (Ind. Eng. Chem., 1927, 19, 312—315).—The oil and vapours in a crank-case are always acid, containing naphthenic and other acids. Running trials were made on cars using gasoline and Pennsylvanian lubricating oil, to ascertain the rate of increase of chamber acidity; with a 6-cylinder car the acidity value attained a maximum at 0·35 mg. of potassium hydroxide per g. of oil, and the dilution a maximum of 19% after running 400 miles, and in other tests the maximum acidity was 0·50 mg. High acidity does not always accompany high dilution, but the acidity is reduced by driving off the diluent. Naphthenic acids were found in the used oil, but not in the condensed water from the crank-case, which contained calcium, magnesium, and iron, and was very corrosive to steel. The acidity values were determined by titration with phenolphthalein as indicator, and the dilution values by the Sligh test in a modified form. H. MOORE.

Determination of phenol. WILLIAMS.—See III.

Ammonium sulphate. WEINDEL.—See VII.

Amyl acetate from gasoline. KOCH and BURRELL.—See XX.

PATENTS.

Plant for washing coal and other minerals by means of liquid streams. A. FRANCE (E.P. 265,077, 18.8.26).—In washing coal with streams of water flowing in launders, the heaviest fractions are made to move with the lowest possible velocity relative to that of the cleaned portions by subdividing the launder by adjustable partitions running parallel to its edges, such partitions being completely submerged in the moving stream. S. PEXTON.

Apparatus for making artificial fuel. R. HAMP (E.P. 264,309, 14.5.26).—Wood refuse is transferred by means of rollers from a hopper to a cylinder in which moves a reciprocating piston. The outlet of the cylinder being of restricted section compresses the fuel in its passage from the cylinder. During the transference of fuel to the cylinder prior to compression, suction is applied to the cylinder through small holes in its side. This ensures complete filling of the cylinder at each stage. S. PEXTON.

Briquetting bituminous coal. S. R. WAGEL, Assr. to LEHIGH COAL & NAVIGATION CO. (U.S.P. 1,623,764, 5.4.27. Appl., 16.6.24. Renewed 31.7.26). —A mixture of bituminous coal with clay and a binder of sulphite liquor, clay, and asphalt is passed through regions of increasing temperature with alternate cooling. C. O. HARVEY.

Preparation of a bituminous composition. L. KIRSCHBRAUN (U.S.P. 1,620,899 and 1,620,900, 15.3.27. Appl., [A], 23.4.21; [B], 25.4.24).—(A) Bentonite is of value as a dispersing agent in the preparation of aqueous bituminous emulsions of the type described in U.S.P. 1,517,075 (B., 1925, 65). (B) Asphalt is emulsified with from 10 to 40% of its weight of bentonite, and dried. The product is infusible, and of value as a waterproofing and roofing material. T. S. WHEELER.

Manufacture of agglomerated adsorbent carbon. SOC. DE RECHERCHES ET D'EXPLOITATIONS PÉTROLIFÈRES (E.P. 261,692, 8.12.25. Addn. to E.P. 244,461; B., 1927, 133).—The mechanical strength of the absorbent carbon is improved by lightly agglomerating the powder with a low proportion of organic binder (preferably with admixture of a dehydrating salt such as ferric chloride), drying the shapes moulded from the resulting pasty mess, coating them superficially with the binder (with or without a small proportion of powdered carbon), calcining at 700° in a luted refractory pot, and washing if necessary. C. O. HARVEY.

Means for gas separation. H. DUMARS and W. S. BOWEN, Assrs. to BOWEN-DUMARS POWER CORP. (U.S.P. 1,622,134, 22.3.27. Appl., 28.11.23).—An apparatus for separating from a gas mixture by refrigeration substances which are, respectively, solid and liquid at the same temperature consists of jacketed condensing and conveying tanks containing baffles on which the solid condensate collects, and a brine tank for receiving liquid condensate. C. O. HARVEY.

Distillation of carbonaceous substances. T. M. DAVIDSON, and PATENT RETORTS, LTD. (E.P. 263,933, 5.10.25).—A carbonising kiln, built in the form of a box, and open at the top, is filled with low-grade fuel. The fuel is ignited at the top and partly burnt by the admission of air through ports in the side. Within the charge are distributed perforated tubes, each of which communicates through a cock to an exhausting fan. The air admission and the removal of distillation products are regulated so that the distillation zone moves slowly down the kiln. S. PEXTON.

Distillation of carbonaceous materials. B. MCCORMICK (E.P. 265,046, 7.5.26).—Carbonaceous material is distilled in a continuous vertical retort in a stream of superheated steam. The steam flows countercurrent to the material, which is supported on a rotating helical partition while undergoing treatment, and the primary oils, conserved by the presence of steam, are removed immediately into zones of lower temperature. S. PEXTON.

Conversion of mineral oils and the like of high boiling point into aliphatic hydrocarbons of low boiling point. F. W. TODT and P. SUMPF (E.P. 267,364, 2.6.26).—Oils particularly suitable for engine fuels, and containing a minimum quantity of unsaturated hydrocarbons and no trace of aromatic compounds, are obtained in yields of up to 80% by a cracking process which involves passing vaporised heavy oils through a cylindrical retort lined with chamotte, charcoal, or the like, charged with porous material, *e.g.*, pumice stone, coke, etc., and maintained at a temperature suitable for successful cracking. The operation is carried out in stages, each stage being adapted (by virtue of temperature or duration of treatment) to the cracking of that fraction of the oil or cracking residue with which the retort is fed. C. O. HARVEY.

Conversion of heavy or complex hydrocarbon oils into lighter oils. J. F. DONNELLY (E.P. 243,339, 10.11.25. Conv., 21.11.24).—Hydrocarbon oil is cracked in a pipe coil under sufficient pressure to prevent vaporisation, and is injected (after admixture with cooler oil to

prevent the formation of solid products) into an expansion chamber connected with a reflux condenser from which all but the heaviest fractions may be drawn off for admixture with the heated oil, the vapours passing on to another condenser. C. O. Harvey.

Conversion of higher-boiling hydrocarbons into lower-boiling petroleum hydrocarbons. C. B. Buerger, Assr. to Gulf Refining Co. (U.S.P. 1,623,025, 29.3.27. Appl., 22.7.25).—Oil, cracked by distilling with aluminium chloride, is freed from hydrochloric acid and other foreign matter by condensing the vapours with water brought into contact with them. C. O. Harvey.

Production of lower-boiling distillates from higher-boiling petroleum hydrocarbons. A. J. H. Haddan. From Gulf Refining Co. (E.P. 267,386, 24.7.26).—50—75% of an oil is cracked to produce gasoline and kerosene containing less sulphur and more saturated hydrocarbons than normal gas oil by distilling continuously with 1—3% of aluminium chloride, and removing the vapours as they are produced, preferably until at least 75% of the still charge has passed over. C. O. Harvey.

Conversion of heavy into light hydrocarbons. C. Ab-Der-Halden (F.P. 610,448, 8.5.25).—The heavy hydrocarbon mixed with steam is passed through molten metal, the water being thereby decomposed into its elements at a temperature at which the hydrocarbon vapour is cracked. A. B. Manning.

Treatment of hydrocarbon oil. H. M. Lasher, Assr. to Kansas City Gasoline Co. (U.S.P. 1,622,453, 29.3.27. Appl., 16.11.20).—The oil is heated in one chamber and cracked by spraying over a fixed catalyst situated above the bottom of a second chamber so as to provide a quiet zone below the catalyst where uncracked oil and carbon may collect, the former being returned to the heating chamber. The lighter cracked products are withdrawn from above the catalyst. C. O. Harvey.

Cracking oils and tars. Soc. Luxembourgeoise des Hydrocarbures, and F. Brimeyer (E.P. 267,776, 20.7.26).—Oils are vaporised and cracked under their own pressure by continuous passage through a heated chamber, and, after condensation, the more volatile fractions are re-evaporated by subjection to the heat of the cracked vapours. After passage through a superheater, the vapours enter a hydrogenating tower maintained at a temperature not above 300°, mix with a stream of hydrogen sulphide (prepared by the action of hydrogen on a metal sulphide), and pass through a metallic mass which liberates nascent hydrogen and thus effects hydrogenation. C. O. Harvey.

Manufacture of benzol and like aromatic hydrocarbons. I. W. Henry (E.P. 267,359, 28.5.26).—Aromatic hydrocarbons are produced from carbonaceous materials (*e.g.*, powdered bituminous coal with or without admixture with limestone) by heating in a retort and subjection to a high-frequency oscillating electro-magnetic field in the presence of hydrogen. The carbon particles suspended in the gas become ionised and react, yielding enriched hydrocarbon gas. C. O. Harvey.

Manufacture of [anti-knocking] liquid fuels. I. G. Farbenind. A.-G. (E.P. 252,019, 10.5.26. Conv., 8.5.25. Addn. to E.P. 226,731; B., 1925, 163).—The anti-knocking effect of iron carbonyl is enhanced and the quantity necessary reduced by adding to benzine containing more than 0·2 g./gal. of the carbonyl a non-metallic organic compound (other than an alcohol) containing oxygen or nitrogen (ketones, aniline, nitrobenzene, etc.), with or without the addition of an organic halogen compound (*e.g.*, halogenides of aliphatic or aromatic hydrocarbons). C. O. Harvey.

Motor fuels. Deutsche Petroleum-A.-G., and E. H. Riesenfeld (G.P. 436,945, 25.2.25).—A motor fuel is made by mixing alcohol and benzine, and adding chlorinated rape oil. This addition ensures uniform combustion in the motor, and homogeneous mixing of the alcohol and benzine. A. B. Manning.

Purification of benzene, benzine, and similar motor fuels. Benzol-Verband G.m.b.H. (G.P. 436,944, 2.2.26).—The oils are freed from sulphur by being brought into contact with amalgams of the heavy metals, in the form of amalgamated metal shavings, wire, or netting, at the ordinary or at higher temperatures. A. B. Manning.

Obtaining light oils by washing gases. I. G. Farbenind. A.-G., Assees. of A.-G. für Anilin-Fabr. (E.P. 254,287, 1.6.26. Conv., 25.6.25).—Light oils are removed from coal and natural gas by washing with the oil dissolved from mineral oils by extracting them with liquid sulphur dioxide (Edeleanu process), which takes up 6% of its weight of benzene as compared with 3% taken up by tar or petroleum oil, and which shows no tendency to thicken. C. O. Harvey.

Refining oils. H. O. Parker, Assr. to Mathieson Alkali Works (U.S.P. 1,622,879, 29.3.27. Appl., 22.1.25).—The oil is passed through aqueous hypochlorite solution containing excess of caustic alkali, and chlorine is introduced into the solution near the point of entry of the oil, which thereby comes in contact first with that portion of the solution containing the highest concentration of hypochlorite. C. O. Harvey.

Method of treating petroleum. R. Cross (U.S.P. 1,623,018, 29.3.27. Appl., 26.4.24).—A minor flow of oil is treated with chlorine, submitted to the action of light, and combined with the major flow. C. O. Harvey.

Separation of hydrocarbon fractions. D. Pyzel, Assr. to Simplex Refining Co. (U.S.P. 1,622,737, 29.3.27. Appl., 23.8.23).—The partial vapour pressure of a portion of a mixture of hydrocarbons is reduced by injecting vapour having a different vapour tension (heat being supplied from the remainder of the mixture which is maintained under its initial vapour tension), and the mixture of vapour is removed. C. O. Harvey.

Removal by centrifuging from fluid hydrocarbons of the solid hydrocarbons precipitating at low temperatures. Bergedorfer Eisenwerk A.-G. (E.P. 267,038, 26.10.26. Conv., 16.8.26).—In a process for the centrifugal separation of paraffins frozen out from lubricating oils etc. the difference in sp. gr. necessary for successful separation is imparted to the

mixture of oil and paraffins by adding a heavy solvent (*e.g.*, trichloroethylene, carbon tetrachloride, etc.) for the liquid hydrocarbons instead of adding gasoline, as is common practice. Only relatively small proportions of these heavy solvents are required, and the paraffins travel to the centre of the bowl, where they are removed along with some of the fluid hydrocarbons, the viscosity being kept sufficiently low, if necessary by the addition of a low-viscosity hydrocarbon. C. O. HARVEY.

Condensation of hydrocarbons. E. W. ISOM and J. E. BELL, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,623,790, 5.4.27. Appl., 10.9.23).—The hot vapours are injected into a large volume of cooled liquid condensate, circulation being maintained by the incoming vapours. C. O. HARVEY.

[Liquid] fuels. J. F. P. DE LA RIBOISIÈRE (E.P. 244,797, 17.12.25. Conv., 17.12.24).—See U.S.P. 1,558,967 ; B., 1926, 6.

Removal of water from peat and the like. O. SÖDERLUND, T. BOBERG, and N. TESTRUP, Assrs. to TECHNO-CHEMICAL LABORATORIES, LTD. (U.S.P. 1,624,769, 12.4.27. Appl., 30.9.24. Conv., 7.11.23).—See E.P. 228,628 ; B., 1925, 274.

Coal drying. O. W. RANDOLPH (U.S.P. 1,623,553, 5.4.27. Appl., 24.12.23).—See E.P. 226,785 ; B., 1925, 976.

Recovery of sulphur compounds of ammonia in the form of salts from gas mixtures containing sulphuretted hydrogen and ammonia. F. SIEMENS A.-G., and H. BÄHR (E.P. 268,024, 23.12.25).—See F.P. 609,931 ; B., 1927, 252.

Production of a porous mass for storing explosive gases. G. DALEN, Assr. to AMERICAN GAS-ACCUMULATOR Co. (U.S.P. 1,623,169, 5.4.27. Appl., 14.8.22. Conv., 16.8.21).—See E.P. 203,255 ; B., 1923, 1060 A.

[Discharging device for] washing and separating apparatus for coal and other minerals. E. DELCUVELLERIE (E.P. 265,112, 16.11.26).

[Burner for] combustion of pulverised fuel in furnaces. L. GROTE (E.P. 264,563, 15.10.25).

Coal distillation (E.P. 266,402).—See I.

Pulverising mill (E.P. 267,426).—See I.

Asphaltic road material (U.S.P. 1,620,813).—See IX.

Bituminous mixtures (E.P. 267,317).—See IX.

Ionising retort (E.P. 267,358).—See XI.

Production of changes in dielectric carbon compounds (U.S.P. 1,621,143).—See XI.

III.—TAR AND TAR PRODUCTS.

Manufacture of pure naphthalene. A. VON SKOPNIK (Chem.-Ztg., 1927, **51**, 211—212).—A suitable tar-oil fraction may contain 40% of naphthalene, but it is desirable to re-distil this in order to obtain the naphthalene with a suitable crystalline structure. The re-distilled oil is subjected to slow crystallisation in vessels of 13 cub. m. capacity, the oil drawn off, and the crystals drained on stages beneath the crystallisers. They then pass through a breaker and are elevated, one third to a melting pan and two thirds to a mixing vessel where the melted portion is reincorporated with the rest. The whole is passed into the press at 50—60°, and the pressure, at first low, is gradually raised to 300 atm. The pressed naphthalene (m.p. 79·0—79·1°) is broken and passed into melting pans. The melted naphthalene is washed with 1% of sulphuric acid (*d* 1·70) to remove moisture and then with 3% of acid (*d* 1·84). A sample of the washed naphthalene after distillation should give no red coloration with sulphuric acid. After water and alkali washes, the product should have m.p. 79·3—79·4°. It is subjected to vacuum distillation with indirect steam in a still having a high column, in presence of a little caustic soda (*d* 1·14). The temperature at the top of the column should be 135—150°, and that of the condensers 105—120°, the latter being maintained with the aid of steam. Tests of the distillate are taken, and when the initial impurities are drawn off the m.p. rises sharply to 79·9°. The pure liquid naphthalene is run into iron crystallising pans in a closed chamber, the cocks being controlled from outside on account of the naphthalene vapour. The aqueous fore-runnings and the resinous still residue are returned to the process. The refined product should give no coloration on melting with an equal volume of sulphuric acid or after remaining 2 hrs. on a watchglass over concentrated nitric acid. If the pressed naphthalene is washed with monohydrate (100% H_2SO_4) the final distillation may be omitted. The product is, however, not quite so good, and the working losses are greater. C. IRWIN.

Pitch softening points. R. G. W. EADIE (J.S.C.I., 1927, **46**, 109—111 T).—Softening points by the Kraemer and Sarnow, ring and ball, and ½ inch cube-in-air methods are given for three grades of vertical-retort tar pitch, along with values for specific gravity, volatile matter, and free carbon content. A simple apparatus for determining softening points is described which gives values corresponding closely with those of the Kraemer and Sarnow and ring and ball methods.

Rapid determination of phenol in ammonia liquor and other solutions. R. D. WILLIAMS (Ind. Eng. Chem., 1927, **19**, 530—531).—In the determination of phenols by their quantitative conversion into bromophenols, special care must be exercised to free the sample from impurities likely to react with bromine. Precise experimental details are given of a method involving the removal of cyanide as cyanate by adding a few drops of ammonium polysulphide, the expulsion of ammonia by boiling with an excess of sodium hydroxide, and the removal of sulphide by oxidation with hydrogen or sodium peroxide, or preferably by precipitation as lead sulphide by means of lead oxide or carbonate. The method may be used with equal facility for determining phenols in ammoniacal liquor, ammonia still waste, benzene containing phenols, and sodium phenoxide solutions equivalent to a wide range of sodium hydroxide and phenol concentration. E. HOLMES.

Estonian shale oil. KOGERMAN.—See II.

Preparation of 2-hydrindone from coal tar. WALTERS.—See IV.

Patents.

Recovery of phenols from ammoniacal liquor or technical effluents. Zeche M. Stinnes, Assees. of F. Ulrich (G.P. 436,522, 14.3.25. Conv., 3.2.26).—Phenols are recovered from ammoniacal or other liquors by extraction with a mixture of benzene or its homologues with the bases from a low-temperature or coke-oven tar. Tar oils from which the phenols have been removed, but which still contain the bases, may be used for the purpose, or the bases themselves may be used alone. Benzene containing 20% of pyridine or quinoline, or preferably such of their homologues as are only slightly soluble in water, extracts phenol quantitatively from an ammoniacal liquor, from which benzene alone removes only 65%. The efficiency of the extraction is due to the chemical action of the bases; the benzene acts merely as a diluent, and may be replaced by benzine or other neutral oil. A. B. Manning.

Manufacture of benzol etc. (E.P. 267,359).—See II.

Purification of benzene (G.P. 436,944).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Analysis of mixtures of the isomeric toluidines. H. H. Evers and N. Strafford (J.S.C.I., 1927, **46**, 114—117 T).—The existing methods for the determination of *p*-toluidine in *o*-toluidine are reviewed and shown to be of limited accuracy. The determination of *m*-toluidine is effected by titration with standard bromate (Callan and Henderson, *ibid.*, 1922, **41**, 161 T). The *meta*-isomeride absorbs 3 atoms of bromine per molecule, whereas each of the remaining isomerides absorbs only 2 atoms; hence the proportion of *meta* can be calculated. *p*-Toluidine is determined from the setting point of the sample to which pure *p*-toluidine has been added in known amount; from the data given, the proportion of this isomeride can readily be calculated. In such mixtures replacement of a portion of the *ortho*- by *meta*-toluidine has no effect on the setting point obtained, hence the method is specific for determination of the *para*-isomeride.

Volumetric analysis of Malachite Green with titanous sulphate and titanous chloride solutions. H. Wilkinson and A. G. Tyler (J. Soc. Dyers Col., 1927, **43**, 116).—Setoglaucine, Malachite Green base, oxalate, and double zinc chloride are decolorised gradually by titration with dilute titanous sulphate solution in 4*N*-sulphuric acid in presence of Rochelle salt. With titanous chloride they are decolorised after the addition of a few c.c. of the reagent, but the colour returns in a few moments, disappearing and returning with each addition of the solution until the end-point is reached, when the colour no longer reappears (cf. Knecht and Hibbert, "New Reduction Methods in Volumetric Analysis," 2nd ed., p. 107). This latter behaviour is shown by Fast Green J.J.O. (sulphate) with both titanous chloride and sulphate. Acid Green G is decolorised gradually with both reagents. E. H. Sharples.

Preparation of 2-hydrindone from coal tar. L. S. Walters (J.S.C.I., 1927, **46**, 150—152 T).—The following method is recommended for the preparation of 2-hydrindone from the "middle" oil fraction of Woodall-Duckham vertical-retort tar. The fraction 175—185° is re-distilled and, after washing with 10% sodium hydroxide solution and 10% sulphuric acid, the bromine value of the dry fraction, 176—182°, is determined. 2 g. of the oil are dissolved in 10 c.c. of carbon tetrachloride in a stoppered flask and kept in the dark for 1 hr. after adding 25 c.c. of 10% bromine in carbon tetrachloride. Excess of 10% potassium iodide solution is added and the iodine titrated with 0·1*N*-sodium thiosulphate, a blank titration being performed on the bromine solution. Three fourths of the total bromine absorption is calculated to indene (1 mol. bromine = 1 mol. indene), and the indene fraction is then heated at 150—160° with the amount of sodium calculated from this figure (at 110—120° if sodamide is employed), 2% of aniline being added as catalyst. The oil is decanted from the sodium-indene, the latter is crushed, added to 10 times its weight of ice-water, and distilled in steam. The oil is separated from the distillate, washed with 1% sulphuric acid, and converted into 2-bromo-3-hydroxyhydrindene by mixing with 10—15% of kieselguhr, emulsifying with 10 vols. of water, and adding slight excess of 10% bromine solution in 10% potassium bromide (cf. Read and Hurst, J.C.S., 1922, **121**, 2550). The bromo-hydroxy-compound is refluxed for ½—1 hr. with 50% alcoholic potassium hydroxide (1 mol.), the alcohol distilled, and the residue refluxed 10—15 min. with excess of 20% sulphuric acid. On rapid steam distillation of this mixture, 2-hydrindone, m.p. 57—58°, is obtained. The bromo-hydroxy-compound cannot be prepared direct from the crude indene fraction. R. Brightman.

Determination of carbon dioxide in carbonates [in dyestuffs]. T. Callan (Analyst, 1927, **52**, 222; cf. B., 1927, 42).—Hepburn's modification of Van Slyke's method of determining carbon dioxide in carbonates has been found useful with dyestuffs. The dry dyestuff is first wetted with 1—2 c.c. of alcohol to prevent frothing, and the reaction was found to be complete in 6 hrs. D. G. Hewer.

Patents.

Preparation of azo dyes. I. G. Farbenind. A.-G., Assees. of H. Eichwede (G.P. 432,426, 11.4.24).—Diazotised amines or aminoazo compounds are coupled with monoazo dyes (or the corresponding ψ-azimines) in which the second component contains at least one hydroxyl, in addition to the auxochromic group, capable of further coupling; sulphonic and carboxylic groups are excluded from all components. The dye 1 : 7-aminonaphthol → resorcinol is coupled with *p*-nitrodiazobenzene (2 mols.); the product, reduced and tetrazotised, gives a deep black when coupled on the fibre with 2 : 3-hydroxynaphthoic anilide. The dye from 2 : 7-aminonaphthol diazotised and coupled acid with 2 : 7-aminonaphthol, is developed on the fibre with diazotised methyl 4-nitro-2-aminobenzoate for a fast yellow-brown. C. Hollins.

Diazo preparations. I. G. Farbenind. A.-G., Assees. of K. Schnitzspahn (G.P. 430,621, 13.2.25).—A mixture of the mineral acid salt of a diazotisable amine, with or without excess of mineral acid or of acid salt, a diluent, and a dry nitrite, remains stable and easily wetted even after long keeping at 60—65°. C. Hollins.

Manufacture of vat dyes and intermediates. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (E.P. 267,177, 12.10.25). — *Chloro-2-methylphenylthioglycollic acid*, m.p. 104°, prepared from 6-chloro-*o*-toluidine, is converted by means of phosphorus trichloride and aluminium chloride into *6-chloro-3-hydroxy-7-methylthionaphthene* [6-chloro-7-methylthioindoxyl], m.p. 102—103°, volatile in steam, which condenses with *p*-nitrosodimethylaniline to form the p-*dimethylaminoanil*, m.p. 256—257°, of *6-chloro-7-methylthiosatin*, m.p. 154° (obtained by hydrolysis of the anil with sulphuric acid). The thioindoxyl is oxidised by air to the *thioindigo* (carmine red on cotton from a greenish-yellow vat), or may be condensed with acenaphthaquinone (for orange), isatin α-anil (violet), 5 : 7-dibromoisatin (red-brown), α-naphthathioisatin (bordeaux-red), bromo-β-naphthathioisatin (from 5-bromo-β-naphthylamine, brown), or with the *p*-dimethylaminoanils of 6-chloro-4-methylthioisatin (bluish-red), 6-ethoxythioisatin (bluish-scarlet), thioisatin (red), 6-chloroisatin (red), and 4-chloro-ββ-naphthathioisatin (violet). C. HOLLINS.

Manufacture of diamino- and aminohydroxydiaryl sulphones from sulphinosalicylic acids and quinoneimines. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 267,366, 7.6.26).—Intermediates for indamines, indophenols, and chromable azo dyes are prepared by condensing an *o*-hydroxycarboxysulphinic acid with a quinone mono- or di-imine, or with a *p*-diamine or *p*-aminophenol and an oxidant. The diamines are readily acylated in the amino-group remote from the sulphone grouping. 5-Sulphinosalicylic acid is condensed with *p*-benzoquinonedi-imine (or with a mixture of *p*-phenylenediamine hydrochloride and ferric chloride) to give 2 : 5-*diamino-4′-hydroxy-3′-carboxydiphenyl sulphone* in 85—90% yield. 2 : 5-Diaminoanisole or -phenetole or *p*-aminodimethylaniline may be used in place of the *p*-phenylenediamine, and 5-sulphino-*o*-hydroxytoluic acid in place of the salicylic derivative. 5-Sulphinosalicylic acid is also condensed with a mixture of ferric chloride and *p*-aminophenol, giving 2-*amino-5 : 4′-dihydroxy-3′-carboxydiphenyl sulphone*; the 4 : 5′-*dimethyl* derivative is similarly prepared from 5-sulphino-*o*-hydroxytoluic acid and 5-amino-*o*-cresol. The monoamines may be diazotised and coupled with resorcinol for yellow dyes, or with R-acid for red dyes. C. HOLLINS.

Manufacture of condensation products of the anthraquinone series. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 252,029, 11.5.26. Conv., 11.5.25). — 3-Aroylbenzanthrones, when fused with alkali, give good yields of compounds of the dibenzpyrenequinone type. Benznaphthapyrenequinone, obtained in this way from 3-α-naphthoylbenzanthrone, m.p. 244—245° (the condensation product from benzanthrone-3-carboxylic chloride and naphthalene), dyes cotton brownish-orange from a red vat. C. HOLLINS.

Manufacture of [indigoid] intermediates and dyes of the anthraquinone and anthracene series. SOC. CHEM. IND. IN BASLE (E.P. 249,489, 19.2.26. Conv., 18.3.25. Addn. to E.P. 210,413; B., 1925, 66).—1(2)-Halogenoanthraquinone-2(1)-carboxylic acids are treated with sodium hydrogen sulphide to give the mercaptan, which is reduced to the mercaptoanthracenecarboxylic acid, and then condensed with halogenoacetic acid to give the thioglycollic-carboxylic acid, which on cyclisation with acetic anhydride and sodium acetate gives 1 : 2- or 2 : 1-anthrathioindoxyl. Further, 1-carboxy-2-anthraquinonylthioglycollic acid, prepared from 2-halogenoanthraquinone-1-carboxylic acid and thioglycollic acid, or from 2-mercaptoanthraquinone-1-carboxylic acid and halogenoacetic acid, is cyclised to anthraquinone-2 : 1-thioindoxyl, or is reduced to 1-carboxyanthryl-2-thioglycollic acid. The thioglycollic acids and thioindoxyls so prepared are condensed with isatins, thioisatins, acenaphthaquinone, β-naphthaquinone, etc., or are oxidised to symmetrical thioindigos. The dyes may finally be halogenated. The following compounds are described: 1-mercaptoanthraquinone-2-carboxylic acid, the 2 : 1-isomeride, and the corresponding anthracene derivatives; 2-carboxy-1-anthrylthioglycollic acid, the 1 : 2-isomeride, and the corresponding thioindoxyls (the 1 : 2-thioindoxyl has m.p. 200°); 1-carboxyanthraquinonyl-2-thioglycollic acid and the corresponding thioindoxyl; vat dyes by oxidation of 2 : 1-anthrathioindoxyl (yellowish-bordeaux on cotton) and the 1 : 2-isomeride (yellowish-brown), by oxidation of 1-carboxy-2-anthraquinonylthioglycollic acid (brownish-grey), by condensation of 2-carboxy-1-anthrylthioglycollic acid with isatin-α-anil (greyish-violet), and by condensation of 1-carboxy-2-anthraquinonylthioglycollic acid with acenaphthaquinone (brownish-orange). Eleven other condensations for vat dyes are tabulated, the colours on cotton being brown or bordeaux, except in the case of the product from 1 : 2-anthrathioindoxyl and the *p*-dimethylaminoanil of 2 : 3-anthrathioisatin, which gives yellowish-green dyeings. C. HOLLINS.

Manufacture of tetrahalogenated 4 : 4′-dimethylthioindigotins. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 254,340, 28.6.26. Conv., 27.6.25).—Bright reddish-violet vat dyes, fast to boiling, are obtained by chlorination or bromination of 6 : 6′-dichloro- (or bromo-)4 : 4′-dimethylthioindigotin, preferably in chlorosulphonic acid or nitrobenzene in the presence of a little iodine. The halogen enters the 5 : 5′-positions. C. HOLLINS.

Manufacture of hexasubstituted thioindigotins. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 254,743, 2.7.26. Conv., 2.7.25. Addn. to E.P. 254,340; preceding abstract).—The method of the prior patent is applied to the production of 5 : 5′-dichloro- and 5 : 5′-dibromo-derivatives of 4 : 4′ : 6 : 6′-tetrahalogeno-, 4 : 4′ : 6 : 6′-tetramethyl-, and 4 : 4′-dihalogeno-6 : 6′-dimethyl-thioindigotins, which are violet vat dyes for cotton. C. HOLLINS.

Manufacture of [azo] dyestuffs. F. STRAUB and H. SCHNEIDER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,624,637, 12.4.27. Appl., 6.3.25. Conv., 20.3.24).—See E.P. 231,149; B., 1925, 875.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Effect of alkalis on wool. II. Effects of sodium and potassium compounds and of temperature. H. C. CHAPIN (Proc. Amer. Assoc. Textile Chem. Colorists,

1927, 42—46).—In solutions of equimolecular concentration, sodium and potassium carbonates produce an equal weakening effect. The effect of sodium or potassium soap is small compared with that of the hot water in which it is dissolved, and much smaller than that of $0 \cdot 1N$-sodium or potassium carbonate solution. The weakening effect of alkali carbonate solution may be reduced by the simultaneous presence of a soap. A rise of 1° in scouring temperature near 50° has the same weakening effect as an increase of 25% in carbonate concentration near $0 \cdot 2N$. CHEMICAL ABSTRACTS.

Dehydration of sulphite spirit with quicklime. E. SCHLUMBERGER (Papier-Fabr., 1927, 25, 180—183).—A method depending upon the elevation of the critical separation temperatures of alcohol–petroleum mixtures by the addition of small quantities of water was used to investigate the dehydration of liquid and gaseous sulphite spirit by lime. A curve for the critical separation temperatures of a series of alcohol–petroleum–water mixtures of known water content was first obtained, and similar temperatures observed for sulphite spirit mixtures then gave directly from the curve the water content of the spirit. Impurities present in the latter, *e.g.*, aldehyde etc., do not affect the measurement. With liquid spirit of 94—96% alcohol content, dehydration was carried out at different temperatures and corresponding pressures in an autoclave, the time in hrs. for the change of a 94% into 98·5% spirit being taken as a measure of the dehydration velocity. A rise in temperature of 10° increases this velocity 2—3-fold, and it is of secondary importance whether the lime is in the form of powder or coarse lumps, or whether the mixture is stirred or not. It is suggested that the dehydration velocity in the heterogeneous system $\text{lime}_{\text{solid}}$–$\text{alcohol}_{\text{liquid}}$ is dependent not upon diffusion (stirring, and the surface development of the lime), but upon a slowly proceeding chemical reaction—apparently the decomposition of an alcohol hydrate. The lime is the more effective the less its water content, *i.e.*, the better it is burnt, marble lime being somewhat less efficient than equally well burnt, but less dense, mortar lime. The most favourable proportion for the dehydration is 125% of the theoretical amount of lime necessary. As less than 1% of alcohol remains in the lime before the combined water passes over, the distillation yield is about 99%. For gaseous spirit, with lime heated at 100°, contact for 15 sec. suffices to dehydrate a spirit from 6% to 0·5% water content. In this case, however, the lime increases in volume about 5-fold through slaking; further, it disintegrates into a very fine powder which is carried over during the distillation, and requires several days to settle out. The advantage of this method consists in the possibility of a continuous process, and still more in the saving of the total heat of vaporisation of the alcohol, which for 1 kg. amounts to about 270 kg.-cal. The disadvantage lies in the increased space required for treatment in the gaseous state.
B. P. RIDGE.

PATENTS.

Preparation of plant fibres, particularly jute, resistant to washing. G. G. GUARNIERI (F.P. 611,289, 16.2.26).—Fibrous materials are immersed in a bath containing enzymes capable of rapidly attacking glutinous impurities, and are then bleached with an alkali hypochlorite, soured, and thoroughly washed.
A. J. HALL.

Production of fine, soft viscose fibres with good physical properties. I. G. FARBENIND A.-G., Assees. of H. LUMMERZHEIM, J. HUBER, and P. ECKERT (G.P. 436,792, 9.8.23).—Highly dispersed emulsoids, preferably of vegetable origin, are added to the liquor for dissolving the xanthate. L. A. COLES.

Preparation of aqueous emulsions of substances insoluble in water. CHEM. FABR. POTT & CO. (Swiss P. 115,701, 28.4.24. Conv., 9.7.23).—Aqueous emulsions of such substances as tetralin and *cyclo*hexanol which are useful agents in the washing and scouring of animal and vegetable raw textile materials, are prepared by means of small quantities of at least one emulsifying agent, *e.g.*, water-soluble sulphonic acids (or their salts) of substituted aromatic hydrocarbons (especially naphthalene derivatives), such as the sodium salt of propylnaphthalenesulphonic acid. A. J. HALL.

Production of patterned weaving effects. HEBERLEIN & CO. A.-G. (E.P. 262,477, 6.12.26. Conv., 5.12.25).—Fabric is woven from both ordinary and nitrated vegetable fibres, treated locally with an alkali, and subsequently heated or steamed, the nitrated fibres being thus removed from the treated parts. Alternatively, the material is printed with a resist or a denitrating agent, or printed with a resist, denitrated, and the resist removed before being passed through an alkaline solution. Finally the whole fabric is made insensitive to subsequent alkaline treatment by being denitrated.
B. P. RIDGE.

Mercerising vegetable fibres. I. G. FARBENIND. A.-G., Assees. of L. LÖCHNER (G.P. 433,733, 16.9.24).—The tenacity of the fibres is increased 40—50%, and they are not turned yellow, by treating them, before or after spinning, at a low temperature, with a hypochlorite mercerising bath of d 1·162. L. A. COLES.

Manufacture of hollow artificial textile fibres. W. J. TENNANT. From J. ROUSSET (E.P. 267,187, 11.11.25. Cf. E.P. 189,973; B., 1923, 91 A).—The usual cellulose solutions (*e.g.*, cellulose acetate in a mixture of acetone and alcohol) are spun into a cell containing air at a temperature substantially higher than the b.p. of the solvent used, a comparatively high rate of flow of this air being maintained in order to produce an active superficial evaporation from the filaments as they emerge from the nozzle. B. P. RIDGE.

Manufacture of a non-hygroscopic pulverulent product from sulphite-cellulose waste liquor. I. G. FARBENIND. A.-G., Assees. of A.-G. FÜR ANILIN-FABR. (E.P. 250,956, 15.4.26. Conv., 15.4.25).—The total solid content of the liquor is obtained in a dry non-hygroscopic form either by evaporating the liquor at a temperature above 100° (*e.g.*, on a drying drum at 140°) or by first heating it at a temperature above 100° and then evaporating it. Maximum stability of the product in air is obtained by subsequently heating it for a short time at a high temperature (*e.g.*, at 180°).
B. P. RIDGE.

Manufacture of cellulose. H. WALKER (E.P. 267,226, 12.12.25 and 19.7.26).—Cellulose pulp is obtained from

the leaves and stems of unripe potato plants (or other solanaceæ) by alternate freezing and thawing alone, or by such treatment followed by open or pressure boiling with water or sodium hydroxide solution.

B. P. RIDGE.

[Manufacture of] artificial silk from nitrocellulose. J. DELPECH (Addn. No. 30,982, 6.10.25, to F.P. 561,614).—Pyroxylin is dehydrated before conversion into spinning solutions. A. J. HALL.

Bleaching cellulose acetate. R. BAYBUTT and E. S. FARROW, JUN., Assrs. to EASTMAN KODAK Co. (U.S.P. 1,623,519, 5.4.27. Appl., 28.10.25).—Cellulose acetate is bleached by bringing ozone into active contact with it. W. G. CAREY.

Production of artificial leather. R. CLAVEL (F.P. 611,401, 30.5.25).—Cellulose esters are treated with aromatic hydroxy-compounds and with metal compounds. *E.g.*, cellulose acetate is successively treated with 1% tannin solution at 70—80°, washed, treated with 3% ammoniacal bismuth nitrate solution for 1 hr., the temperature being gradually raised to 65°, steeped in ferric chloride solution, washed with dilute hydrochloric acid, dyed, dried, calendered, and printed. Zinc, silver, and iron salts can be used instead of bismuth nitrate. L. A. COLES.

Production of waterproof materials from textiles, paper, and the like, by impregnation with metal chlorides. A VOHL & Co., A.-G. (G.P. 433,983, 8.8.25. Addn. to G.P. 377,659; B., 1923, 971 A).—The necessity for heating the material on rollers to effect partial hydrolysis of the cellulose to amyloids, as described in the earlier patent, is avoided by using a bath of *d* 1·616—2·244 maintained at 60—90°. L. A. COLES.

Production of films not sensitive to electrical action. PATHÉ CINÉMA (ANCIENS ÉTABL. PATHÉ FRÈRES) (F.P. 611,136—7, 26.5.25).—Nitrocellulose films are coated on the one side with the photographic emulsion, and on the other with a mixture of (A) cellulose nitroacetate, or (B) cellulose benzoate, with a plasticising agent. L. A. COLES.

Altering the viscosity of materials containing cellulose. H. P. BASSETT (U.S.P. 1,620,938, 15.3.27. Appl., 19.12.23).—Cellulose fibre is heated with 0·05—1% sulphuric acid at 20—100°, washed, treated with 0·1—2·5% sodium hydroxide solution, and washed again. The product when dissolved in the usual solvents for cellulose gives solutions of high or low viscosity, according as the weaker acid and the lower temperature, or the stronger acid and the higher temperature, have been employed. T. S. WHEELER.

Waterproofing textile and other materials. C. J. MORETON (U.S.P. 1,625,672, 19.4.27. Appl., 22.1.26. Conv., 24.12.25).—See E.P. 262,605; B., 1927, 165.

Manufacture of a cellulose product having the appearance of wool. A. PELLERIN (E.P. 263,727, 12.2.26. Conv., 30.12.25).

Apparatus for boiling or otherwise treating cork and like materials. W. BRIDGES (E.P. 267,652, 11.1.26).

Means for obtaining fibrous materials from the stalks of hemp and like plants. F. B. DEHN. From FABRICORD INC. TRUSTEE (E.P. 267,763, 31.5.26).

[Spinning head] for spinning artificial silk. A. E. O'DELL. From BERLIN-KARLSRUHER INDUSTRIE-WERKE, A.-G. (E.P. 267,727, 12.4.26).

[Paper-]pulp forming machine. C. J. KEENAN, E. P. KENNEDY, and G. H. KIRSCH, Assrs. to ALASKA PULP & PAPER Co. (U.S.P. 1,621,671, 22.3.27. Appl., 9.2.26).

Treatment of glass (E.P. 267,428).—See VIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Progress in the application of vat dyes. F. M. ROWE and C. P. BEAN (J. Soc. Dyers Col., 1927, **43**, 99—105).—A review of the application of vat dyes and an investigation of the properties and application under varying conditions of development on cotton and wool of Soledon Jade Green, Soledon Brilliant Purple RR, and Soledon Yellow G. In dyeing cotton and wool from a Glauber's salt bath more level dyeings are obtained, particularly with wool, by entering the material into the cold bath instead of a warm bath as recommended by the manufacturers. Soledon Yellow G gave the best results when the material, previously boiled out in 2% sodium carbonate solution and rinsed lightly, was entered into the cold dye-bath, which was heated quickly to 50°, then during ½ hr. at 70°, and kept at that temperature for ½ hr. The presence of the trace of alkali prevented any decomposition in the dye-bath, but diminished the rate of exhaustion. The behaviour of cotton and wool dyed with these dyes on development with mineral acids, nitrous acid, acid persulphate, acid ferric chloride, and acid dichromate is described.

E. H. SHARPLES.

Cloth dyeing for rubber-proofing. H. L. HOCKNEY and C. W. BANCROFT (J. Soc. Dyers Col., 1927, **43**, 105—110, and Trans. Inst. Rubber Ind., 1927, **2**, 435—445).—The precautions necessary in the dyeing of cloth for rubber-proofing are discussed. The pronounced tendering effect of traces of mineral acidity in materials to be hot cured and of oxalic acid in the steam cure is emphasised. The presence of minute amounts of metals in the cloth accelerates the degradation by oxidation of the vulcanised rubber, the most common and deleterious being copper. Rubber cured by the cold process is most susceptible to traces of copper, and it is considered that cloth containing 0·01% or more of copper is useless for proofing, and thus those dyeing processes directly employing copper salts and dyeing or dye-making plant containing copper fittings are to be avoided. Manganese is not as dangerous as copper, but it is not advisable to use the permanganate process for bleaching. The presence of grease or oil, especially in presence of a trace of copper, has a very deleterious effect on the physical properties of the rubber, and they should not be present in amounts more than 2%. The effect of the curing processes on the dyes is discussed, and it is considered that it is almost imperative to submit sample dyeings to the actual conditions of proofing. E. H. SHARPLES.

Applications of vat colours in printing. L. SMITH (J. Soc. Dyers Col., 1927, **43**, 110—114).—A

description of the methods employed and the practical difficulties encountered in printing with vat dyes.
E. H. Sharples.

Patents.

Production of fast-coloured discharges on fast dyeings. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 246,183, 19.1.26. Conv., 19.1.25).—Two vat dyes are used, one of which is not fast to formaldehydesulphoxylate in the presence of Leucotrope W, the other being fast to these reagents. The material is dyed with the dischargeable vat dye and then printed with a paste containing the fast vat dye, together with Leucotrope W, formaldehydesulphoxylate, anthraquinone, etc. Suitable pairs of dyes are Indanthrene Orange RRT printed on Indanthrene Olive R; Indanthrene Blue 3G and Indanthrene Yellow G on Indanthrene Red-violet RH; Indanthrene Violet BN on Indanthrene Brown G; Indanthrene Brilliant Blue R on Helindone Fast Scarlet B.
C. Hollins.

Dyeing and printing cellulose esters. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 267,695, 25.2.26).—Sulphamic acids or sulphamates, especially the *N*-sulphonic acids derived from aminoanthraquinones, dye cellulose esters; the dyeings may further be developed with diazo compounds, or may be diazotised and coupled with suitable components. Examples are the sulphamic acids derived from 1 : 4-diaminoanthraquinone (reddish-violet), 1 : 5-diaminoanthrarufin (reddish-blue), 1 : 4-aminomethoxyanthraquinone (bluish-red), 4-aminobenzeneazo-β-naphthol (reddish-orange), 2 : 4-dinitro-4′-aminodiphenylamine (golden-yellow).
C. Hollins.

Protection of animal fibres in mordant dyeing. I. G. Farbenind. A.-G., Assees. of K. Daimler, G. Balle, and F. Just (G.P. 435,899, 21.12.23).—Resinous sulphonic acids obtained from condensation products of aromatic or hydroaromatic hydrocarbons with sulphur chloride, alkyl, aryl, or aralkyl chlorides, alcohols, etc. are added to the mordanting and dyeing baths. An example is sulphonated benzylnaphthalene resin. C. Hollins.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Manufacture of sulphuric acid. The Tenteleff process. P. P. Budnikov (Chem.-Ztg., 1927, **51**, 209—210, 230—232).—The plant and process differ in various details from the Badische process. The burner-gas after dust deposition is passed downwards through a lead cooling tower surrounded by an iron water jacket with cross connexions passing through the tower. The gas leaves at the bottom at 90—120°, and some dilute and impure sulphuric acid is condensed and drawn off. Coke or quartz filters are now not used; the gas is passed through a washer in the shape of a heavy lead bell with water jacket, in which the wash water is changed once daily. It then enters the washing tower, which consists of five superimposed lead cylinders fitted with perforated bells. Contact is further promoted by perforated plates in each section, and the water is changed either intermittently or continuously. In some cases alkali is added to the water in both types of washer to assist in removing chlorine. The gas is then dried in four coke towers of lead, the last three of which are fed with sulphuric acid of increasing concentration. The moisture content of the dried gas, determined by means of phosphorus pentoxide, should not exceed 0·01%. The dried gas enters a cigar-shaped vessel which serves as a reservoir for the compressors and also for the deposition of entrained acid spray. After passing the compressors the gases are freed from oil in two lead-lined iron vessels charged, respectively, with coke and asbestos, and are preheated at 220—240°. The converter consists of a wide upper part containing the principal contact mass of platinised asbestos, and a narrower lower part having 25 perforated plates with further contact masses between them. Conversion averages 96%. Three absorbers in series are used for an output of 10 tons of sulphur trioxide per 24 hrs. These are enamelled cast-iron vessels with four inlet pipes, fed continuously with acid and cooled with water. The last absorber produces monohydrate. The exit gases contain 0·01% SO_3. Alternative forms of some of the apparatus are described and dimensions given. C. Irwin.

Active forms of silicic acid (silica gel) and their adsorptive powers. O. Ruff and P. Mautner (Z. angew. Chem., 1927, **40**, 428—434).—Largely a critical review of the literature. It is suggested that the adsorptive power of a solid depends on three effects: (*a*) an unspecific, purely surface effect, analogous to the adsorption of gases, (*b*) a specific adsorption of substances of molecular dispersity, (*c*) adsorption of colloids, which for very small particles is specific, but for large particles is a kind of filter action. Silica gel adsorbs appreciable quantities of acetic acid from aqueous solution, and also smaller quantities of aniline. In respect of the adsorption of acetic acid from solution in petrol, kieselguhr dried at 350° is superior to ordinary ground quartz or powdered vitrified quartz, but is not so good as silica gel.
R. Cuthill.

Causes of discoloration of ammonium sulphate produced at coking plants. A. Weindel (Brennstoff-Chem., 1927, **8**, 104—107).—The grey colour of "neutral" ammonium sulphate is primarily due to iron sulphide, and to a less extent to sulphides of lead and copper. Arsenic sulphide, being yellow, may be disregarded in this respect. The discolouring effect of tarry substances present in the crude ammonia liquor is negligible. Sulphuric acid made by the contact process is preferable to chamber acid, as it contains less metallic impurities, and the traces of nitrogen oxides in chamber acid increase its corrosive action. Acid sulphate may be neutralised either before or after centrifuging, or in the centrifuge itself. In no case is complete neutralisation attained, the "neutral" salt containing both free acid and free alkali. Neutralisation with ammonia liquor in the centrifuge leads to an undue amount of copper in the mother-liquor, due to corrosion of the copper basket and gauze. The two methods recommended are: neutralisation of the hot salt and mother-liquor, before centrifuging, with gaseous ammonia free from hydrogen sulphide, or the treatment of the centrifuged acid salt with ammonia gas or solution free from hydrogen sulphide. It is for many reasons advantageous to remove the tarry matter from the crude ammonia liquor before

it reaches the still, and this may be done by filtering it through a crushed hydrophobic substance, such as coal dust. W. T. K. Braunholtz.

Volumetric determination of magnesium in magnesium chloride solutions. J. E. W. Rhodes (J.S.C.I., 1927, **46**, 159—160 T).—2·5 c.c. of a solution containing 30—35% of magnesium chloride, or an equivalent amount of a weaker solution, are run into a weighed 100 c.c. flask. The flask is again weighed and filled to the mark with 0·25*N*-baryta solution, shaken, and kept for 4 hrs. 25 c.c. of the supernatant liquor are then pipetted off and titrated with 0·1*N*-hydrochloric acid in presence of phenolphthalein. The first disappearance of colour is the correct end-point. The percentage of magnesium chloride found is usually within 0·15 of that found by gravimetric analysis.

Stability of titanous sulphate solutions in air. H. Wilkinson and A. G. Tyler (J. Soc. Dyers Col., 1927, **43**, 114—115).—A titanous sulphate solution having a fair degree of stability is prepared as follows. 1500 c.c. of 4*N*-sulphuric acid are run into a 2-litre stock bottle, and zinc amalgam is distributed evenly over the bottom. A few pieces of granulated zinc are added to remove the dissolved oxygen, and a layer of solvent naphtha is run over the surface of the liquid. 30 c.c. of commercial titanous sulphate solution are made up to 200 c.c. with 4*N*-sulphuric acid in a conical flask, and a quantity of zinc amalgam is added. Carbon dioxide is passed through the liquid, which is boiled until a clear violet solution is obtained. It is then cooled and decanted into the stock bottle. With simple standardisation with potassium permanganate from time to time such a solution is very convenient for technical purposes, although for accurate work the solution must be kept under an atmosphere of hydrogen, as recommended by Knecht and Hibbert. E. H. Sharples.

Ionic exchange of zeolitic silicates with hydrolysable salts. I. Experiments with permutite. H. Kappen and F. Rung (Z. Pflanz. Düng., 1927, **A8**, 345—373).—The effect upon the exchange acidity shown by a pure sodium and calcium permutite of treatment with various salts has been studied in detail. The results of experiments with ferric chloride, chromic sulphate, and aluminium chloride indicate that it is impossible to obtain a direct exchange of ferric, chromic, and aluminium ions with the bases of the permutite. It is considered that the physical condition of the permutite accounts for this. On the other hand, treatment with ferrous, cupric, and zinc salts gives permutites in which exchangeable ferrous, cupric, and zinc ions are present. The formation of aluminium permutite by treatment with acids was also studied. The discussion of the bearing of these results on problems of soil acidity is deferred until the results of experiments with natural crystalline silicates are available.

C. T. Gimingham.

Carbon dioxide in carbonates. Callan.—See IV.

Toxic action of selenium and tellurium compounds. Stover and Hopkins.—See XVI.

Patents.

Manufacture of nitric acid. C. Toniolo (E.P. 267,721, 27.3.26).—Instead of absorbing oxides of nitrogen from the combustion of ammonia or other source in refrigerated dilute acid circulating in towers in counter-current to the gases (cf. E.P. 121,635; B., 1919, 104), the acid and the gases both enter the towers through the top, the hot gases thereby undergoing sudden cooling by the acid, which is cooled to near zero before entry. The gases instead of the acid may be cooled to considerably below zero, the acid circulating in the absorption towers at a higher temperature, but not so high as to cause dissociation. The refrigeration of the gases may be brought about in towers through which strongly cooled nitric acid circulates in the same direction as the gases. W. G. Carey.

Production of hydrochloric acid and magnesia. Verein für Chem. & Met. Prod. (G.P. 436,241, 11.3.25).—Magnesium chloride or oxychloride is treated with steam in a revolving tube furnace heated both internally and externally. L. A. Coles.

Evolution of hydrocyanic acid [for fumigation]. H. Lehrecke, Assr. to Roessler & Hasslacher Chemical Co. (U.S.P. 1,620,365, 8.3.27. Appl., 20.10.25. Conv., 6.11.24).—Anhydrous zinc chloride absorbs 3 mols. of anhydrous hydrogen cyanide, which is liberated on exposure of the product to the action of moisture. For fumigation it is of advantage to add an excess of zinc chloride or other substance with a large heat of hydration. T. S. Wheeler.

Production of colloidal silicic acid solutions. M. Prätorius and K. Wolf (F.P. 612,486, 8.3.26. Conv., 18.3.25).—Sodium silicate solution is electrolysed in a cell containing at least three diaphragms constructed of non-reacting material, such as wool, asbestos, or alumina, between the two poles. L. A. Coles.

Recovery of titanic acid, iron, and magnesia from titaniferous ores. F. E. Bachman (U.S.P. 1,618,795, 22.2.27. Appl., 18.3.21).—A mineral containing titanium, *e.g.*, ilmenite, intimately mixed with carbon and sodium carbonate is heated in a rotary kiln, and the clinker formed is introduced with carbon into a blast furnace, when metallic iron and a slag are obtained. The slag is heated with water under pressure to dissolve the sodium compounds present, and leave hydrated titanic oxide and magnesium compounds, which are heated with dilute sulphuric acid under pressure at 150°. The residue from this treatment is dissolved in sulphuric acid, treated with titanous chloride to reduce the trace of iron present, and reprecipitated by heating under pressure. Finally, the sulphate present is removed with barium hydroxide, leaving titanic acid. T. S. Wheeler.

Apparatus for the catalytic combustion of mixtures of ammonia and oxygen. I. W. Cederberg (E.P. 244,134, 8.12.25. Cf. E.P. 236,145; B., 1925, 713).—The walls of the apparatus are two hexagonal chrome–nickel steel plates connected to form a gas-tight joint and having jackets for cooling water. The intermediate space is extremely narrow, especially at the centre, so that a gas layer is formed sufficiently thin to prevent explosions, and this space contains a catalyst of platinum gauze or sheet. W. G. Carey.

Synthesis of ammonia. Soc. Chim. de la Grande Paroisse, Azote & Prod. Chim. (F.P. 611,417, 2.6.25).—

The supply of the mixed gases to each unit in apparatus comprising a number of catalyst units in parallel is controlled by regulating the pressure before or behind each unit, or by the provision of a pump having a cylinder for supplying each unit. L. A. Coles.

Manufacture of sodium peroxide. Roessler & Hasslacher Chemical Co. (E.P. 265,124, 7.6.26. Conv., 29.1.26. Cf. E.P. 264,724; B., 1927, 218).—Molten sodium, highly diluted with powdered sodium monoxide, is oxidised to sodium monoxide in a dry atmosphere poorer than air in oxygen preferably below 200° and not above 250°, and the resulting monoxide is oxidised to sodium peroxide in an atmosphere richer than air in oxygen at 200—350°. W. G. Carey.

Recovery of sulphur [from charcoal]. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 267,246, 17.12.25).—Sulphur is recovered from masses containing it, such as active charcoal from the catalytic oxidation of hydrogen sulphide, by passing superheated steam through them in a downward direction at a speed of at least 1 m./sec., and collecting the molten sulphur. The steam is washed with water to free it from sulphur vapour or mist, and is superheated and used again. W. G. Carey.

Eliminating water from liquid sulphur dioxide. Allgem. Ges. für Chem. Ind. m.b.H. (E.P. 261,732, 6.11.26. Conv., 19.11.25).—Water taken up by liquid sulphur dioxide in the refining of hydrocarbons or oils and fats is eliminated by compression and cooling, and at the first stage of the condensation the liquid, which contains all the water, is tapped from a preliminary condenser, the sulphur dioxide therein being re-introduced to the system after distillation to separate the water. W. G. Carey.

Manufacture of nitric acid. C. Toniolo (U.S.P. 1,623,606, 5.4.27. Appl., 9.9.26).—See E.P. 267,721; preceding.

Production of concentrated solutions of alkali cyanides. Deutsche Gold- u. Silber-Scheideanstalt, vorm. Roessler (E.P. 245,152, 23.12.25. Conv., 23.12.24).—See U.S.P. 1,615,208; B., 1927, 218.

Manufacture of siliceous alkaline-earth compounds. A. J. H. Haddan. From Celite Co. (E.P. 268,011, 18.12.25).—See U.S.P. 1,574,363; B., 1926, 487.

Converting hafnium and zirconium phosphates. J. H. de Boer, Assr. to N. V. Philips' Gloeilampenfabr. (U.S.P. 1,624,162, 12.4.27. Appl., 19.6.25. Conv., 13.8.24).—See E.P. 238,543; B., 1925, 881.

Sulphur compounds of ammonia from gas (E.P. 268,024).—See II.

Utilisation of phosphate residues (E.P. 252,367).—See IX.

Zirconium and hafnium (U.S.P. 1,618,960).—See X.

VIII.—GLASS; CERAMICS.

U.S. Government master specification for plastic fire-clay refractories. (U.S. Bur. Standards, Circ. No. 297, Oct. 20, 1926, 5 pp.).—The fire clay, specially adapted for the construction of small furnaces, must contain not more than 65% of total silica calculated on the dry weight, and the softening point shall not be less than 1680°; the water content as delivered shall not exceed 15% calculated on the plastic weight, nor the total linear dry-and-burned shrinkage 4% of its plastic length. B. W. Clarke.

U.S. Government master specification for fire clay. (U.S. Bur. Standards, Circ. No. 298, Oct. 20, 1926, 6 pp.).—Fire clay is divided into two classes, fine and commercial, according to its fineness. Of the former, not more than 4% is retained on a U.S. standard sieve No. 20, and of the latter, 10%. It must have a softening point not more than 60° lower than that of the brick with which it is to be used. B. W. Clarke.

U.S. Government master specification for fire-clay brick. (U.S. Bur. Standards, Circ. No. 299, Oct. 20, 1926, 7 pp.).—Fire-clay brick is divided into the following classes: SH 75, for severe conditions of boiler practice, with a softening point of not less than 1680°; H 75, for general boiler practice; and H 57, for use where resistance to spalling is unimportant, with ability to withstand 15, 12, and 5 quenchings, respectively, without failure. Other classes are:—M 73, for use at moderate temperatures, softening point not less than 1640°; also H 25 and M 7, for silicious brick with deformation under load at high temperatures of less than 3% and 4% respectively. The resistance to spalling, slagging, or high temperatures varies for each class, and is determined in a "simulated service test," in which the brick is built into one wall of an experimental oil-fired furnace of special design, for comparison with standard brick, observation being taken by radiation pyrometers of the inner- and outer-face temperatures of the furnace and a spalling test conducted at the end of the run. B. W. Clarke.

Patents.

Ray-filter glass. I. Frank (U.S.P. 1,615,448, 25.1.27. Appl., 6.7.25).—A glass which is practically colourless in commercial thickness and gives normal transmission in the visible region, but which absorbs the greater portion of the ultra-violet and infra-red rays, is obtained by addition of suitable proportions of ferric oxide and manganese dioxide to the batch. For lenses approximately 1·5 mm. thick, a proportion of 0·00256 pt. by wt. of the batch of the mixture of oxides in the ratio of manganese dioxide to ferric oxide of 3 : 2 is used. A. Cousen.

Readily fusible glasses and enamels. General Electric Co., Assees. of Patent-Treuhand-Ges. für Elektrische Glühlampen m.b.H. (E.P. 267,815, 25.9.26. Conv., 11.5.26).—Glasses with a low coefficient of expansion and without yellow coloration, for coating glass articles by fusion, have the following limiting composition, 60—75% of lead oxide, 10—25% of zinc oxide, and 15—30% of boric oxide, the most suitable formula being 65% of lead oxide, 15% of zinc oxide, and 20% of boric oxide. They may be made opaque by adding up to 2% of arsenic and up to 2% of an ammonium compound. W. G. Carey.

Treatment of glass plates and the like to prevent moisture affecting their transparency. O. Demaret (E.P. 267,428, 1.11.26. Conv., 27.4.26).—A transparent

sheet or film of coagulated or reverted cellulose, previously treated with 50% caustic potash solution at 30°, washed, and dried, is secured by transparent glue to the surface of the glass. B. W. CLARKE.

Moulding silica. BRITISH THOMSON-HOUSTON Co., LTD., Assees. of L. B. MILLER (E.P. 262,110, 23.11.26. Conv., 24.11.25).—Silica is rendered plastic by heating at 1700°, and a moulding member of carbonaceous material at approximately the same temperature is introduced into the mass. To prevent chemical action between the silica and the mould these are removed and cooled rapidly in the air, thereby facilitating the removal of the mould from the solidified silica. B. W. CLARKE.

Treatment of silica articles. BRITISH THOMSON-HOUSTON Co., LTD., Assees. of E. HERZOG (E.P. 264,863, 21.1.27. Conv., 21.1.26).—Articles of vitreous silica, having a rough surface which is to be glazed, are mounted so that they can be rotated and heated in an enclosed space at about 1000°, which is well below the softening temperature, but sufficiently high to prevent cracking during the subsequent process. The articles are then subjected to a fusing temperature from the flames of suitably arranged burners, the region of fusing heat being continuously changed by rotating the article and traversing the burners until the whole surface is glazed. B. W. CLARKE.

Kiln for glass melting and like purposes. G. E. CRAWFORD, Assr. to AMERICAN DRESSLER TUNNEL KILNS INC. (U.S.P. 1,623,779, 5.4.27. Appl., 19.10.23).—A tunnel kiln has an elongated kiln chamber and a movable hearth separated from a portion of the kiln structure by a clearance space at the side of the hearth. A conveyor beneath removes material passing downwards through the clearance space. W. G. CAREY.

Manufacture of raw plate glass. M. BICHEROUX (E.P. 263,847, 29.12.26. Conv., 30.12.25. Addn. to E.P. 182,551).

Stretching glass. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY, ET CIREY (E.P. 257,590, 12.8.26. Conv., 27.8.25.)

[Fritted-glass] filter (U.S.P. 1,620,815).—See I.

IX.—BUILDING MATERIALS.

Aqueous vapour pressure of magnesium oxychloride cement, and the state of the water contained in the latter. T. MAEDA (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1926, 5, 133—140).—The aqueous vapour pressure isotherms of two magnesium oxychloride cements are constructed for 25°, with a view to the elucidation of the state of the water contained in them. This vapour pressure is due, in one specimen of cement containing 33% of water, mainly to the water of crystallisation of the double compound $3MgO,MgCl_2,12H_2O$, whilst in the other, containing 55% of water, it is ascribable chiefly to magnesium chloride solution held by capillarity within the pores of the cement. Cements in which the water is preponderatingly of the former type are brittle, and sweating of the surface of the cement in a moist atmosphere is more marked, this being due either to the lower vapour pressure of its water, or to its more compact surface preventing drops of water from soaking into the interior of the mass. R. TRUSZKOWSKI.

Hardening of magnesium oxychloride cement, and the function of free water, water of crystallisation, and adsorbed water. T. MAEDA (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1926, 141—154).—Measurements of the hardness of magnesium oxychloride cements, using a modified Vicat apparatus, indicate that hardening is a continuation of the setting process, and is due to the further formation of the double salt $3MgO,MgCl_2,12H_2O$. Elimination of water and the absorption of carbon dioxide play only a secondary rôle in this process. A high proportion of solid constituents without sufficient adsorbed water leads to the formation of brittle cements, as does also the use of old specimens of magnesium oxide. Free water is necessary for the plasticity of cement, and water of crystallisation and adsorbed water influence the setting and hardening processes, the latter form of water also serving to reduce the brittleness of the cement. Soft cements are characterised by an unduly high free-water content after hardening. R. TRUSZKOWSKI.

Influence of humidity changes upon the composition of building materials. J. W. MCBAIN and J. FERGUSON (J. Physical Chem., 1927, 31, 564—590).—Data on the sorption of water vapour under varying conditions of humidity have been obtained using the sorption balance of McBain and Bakr (A., 1926, 493) in the case of the following materials: powdered clear quartz crystal, fibres of fused silica, kieselguhr, Filter cel ("treated" diatomaceous earth), Millstone grit (Idle Quarry and Darley Dale), Kentish rag, Portland stone, Mona's Park stone, dolomitic limestone (Gold Coast), chalk (Setch, Norfolk), Stoneycombe stone, Buxton limestone, Meldon granite, and the following bricks:—red rubber, red wire-cut, blue wire-cut, blue Stafford, Fletton, Stock and sand-lime. Quartz, granite, the pure limestones, and highly-burned brick show practically no sorption from an atmosphere saturated with water vapour. Portland stone and Monk's Park stone show initially a slight absorption of water vapour which remains unchanged over a wide range of humidity. The building stones absorb 1% or less, varying with humidity changes, and the amorphous silica, such as kieselguhr, sorbs up to 10% of water at high humidity, the amount varying largely with conditions. The sorption and swelling of sandstone is due to the contained amorphous building material. The hysteresis shown by kieselguhr, Filter cel, and Gatton stone has also been studied. Absorption of methylene-blue and magenta by building materials parallels the absorption of water vapour. Heating at 850° destroys the colloidal matter present in sandstones, which then lose the power of sorbing water vapour or dyes. The inter-grain cement of sandstone sorbs the vapour of water, methyl and ethyl alcohols, chloroform, benzene, and octane according to the size of the molecules concerned. The heats of sorption have been calculated for certain cases. The results of the sorption experiments agree with the conception of micellar linkage advanced in connexion with other work (cf. A., 1926, 351), and on

this basis is advanced the definition that a stone preservative is a substance which in the presence of water is preferentially sorbed by the mineral gel, and remains permanently in combination with it. L. S. Theobald.

Patents.

Apparatus for agitating slurry etc. J. S. Fasting, Assr. to F. L. Smidth & Co. (U.S.P. 1,622,280, 29.3.27. Appl., 5.9.24).—A carrier supported on the walls of a slurry tank, along which it is free to move, is provided with a shaft carrying a series of sprocket wheels which can be rotated, and from which chain loops are suspended within the tank. Weighted air pipes, connected to a supply of compressed air, are supported pivotally from the carrier. B. W. Clarke.

Manufacture of hydraulic cement and products produced thereby. H. S. Spackman (E.P. 244,756, 10.12.25. Conv., 17.12.24).—A charge, consisting of bauxite and limestone in such proportions that the acidic components are approximately equal to the basic components, is heated in a rotary cement kiln in an oxidising atmosphere, producing a clinker which is ground to form a hydraulic cement containing 11% of iron oxide, together with alumina, lime, and silica. B. W. Clarke.

Manufacture of cement. L. Kern (U.S.P. 1,623,876, 5.4.27. Appl., 23.12.25).—A mixture of natural calcareous and siliceous material, after calcination at a temperature too low to cause sintering, is pulverised, slaked with sufficient water to give a mass capable of being moulded, and, after moulding, is hardened under pressure at steam heat and burnt at 400—600°. W. G. Carey.

Production of a porous cementitious article. A. T. Schenck (U.S.P. 1,622,396, 29.3.27. Appl., 4.1.26).—Cement in granulated form is mixed with hydrated lime, aluminium, and warm water, and during the liberation of gas the mixture is moulded and rotated during the setting to form substantially uniform voids in the mass. B. W. Clarke.

Utilising and producing cement from the residue obtained in the reduction of molten crude phosphates. I. G. Farbenind. A.-G., Assees. of M. Platsch (E.P. 252,367, 17.5.26. Conv., 23.5.25).—Cement is obtained by fusing a crude phosphate in an electric furnace with sand and carbon or coke, and, after the phosphorus vapours are expelled, adding calculated amounts of alumina (bauxite or clay) until a homogeneous cement mixture is produced, which is tapped off and ground. W. G. Carey.

Production of gypsum product. L. M. Parkhurst, Assr. to Federal Gypsum Products Co. (U.S.P. 1,620,915, 15.3.27. Appl., 6.5.22).—A mortar for making gypsum products is prepared by mixing calcined gypsum with water and carbon dioxide. W. G. Carey.

Composition capable of being moulded. Michelin et Cie. (E.P. 263,138, 13.12.26. Conv., 19.12.25).—A mixture of 15 pts. of sodium silicate solution and 100 pts. of ground lava is moulded under pressure, dried in air, stoved at 40—50°, and finally baked at 800—900°. The product is resistant to shocks and cold. B. W. Clarke.

Bituminous mixtures for use in road construction and the like. Universal Rubber Paviors (Manchester 1923), Ltd., and A. E. Brown (E.P. 267,317, 12.9.25).—Residual products such as guttapercha pitch or balata pitch are added to bituminous mixtures other than those described in E.P. 246,186 (cf. B., 1926, 276). B. W. Clarke.

Preservation of wood. L. P. Curtin, Assr. to Western Union Telegraph Co. (U.S.P. 1,620,152, 8.3.27. Appl., 24.6.25).—Wood is impregnated with an aqueous solution containing copper sulphate, sodium hydrogen sulphite, sodium carbonate, sodium chloride, arsenious oxide, and sodium fluoride. On exposure to air cupric hydrogen orthoarsenite is deposited on the wood. T. S. Wheeler.

Apparatus for impregnating wood. K. Bubla (E.P. 267,579, 18.11.25).

Manufacture of mastic sheet material. L. Mellersh-Jackson. From F. R. Muller Inc. (E.P. 267,414, 1.10.26).

Bituminous composition (U.S.P. 1,620,900).—See II.

Coating wood etc. (E.P. 252,364).—See XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Influence of atmosphere and temperature on scaling of steel. I. Scaling by air, water vapour, and carbon dioxide. C. B. Marson and J. W. Cobb. **II. Prevention of scaling by hydrogen and carbon monoxide.** H. T. Angus and J. W. Cobb (J.S.C.I., 1927, **46**, 61—72 T).—The production of a scale of oxide on iron or steel in re-heating operations may occur even in an atmosphere containing no free oxygen. Laboratory experiments have been made to ascertain the conditions which determine scale production on a boiler-plate steel (0·2% C) at 900° and 1000°. The weight of scale was measured when samples identical in form and size were exposed to different atmospheres for 3 hrs. At 1000° the weights of scale produced in g./cm.^{2} were: in dry air 0·0635, in 20% steam : 80% nitrogen 0·0166, and in 20% carbon dioxide : 80% nitrogen 0·0024. When both carbon dioxide and steam were present the results were intermediate. Adding carbon monoxide to carbon dioxide retarded but did not stop scaling altogether until 58% of carbon monoxide was present. Scaling by steam was arrested only when 50% of hydrogen was present. The addition of hydrogen to carbon dioxide increased the rate of scaling owing to the formation of steam by way of the "water-gas reaction," the maximum scaling occurring with an initial mixture of 26% H_2 : 74% CO_2. Further addition of hydrogen reduced the scaling, which was completely arrested by 57% of added hydrogen. The addition of carbon monoxide to steam reduced the rate of scaling, but did not stop it until 80% of carbon monoxide was present. It required the addition of 30% of methane to stop the scaling of an atmosphere containing 10% of carbon dioxide and 10% of steam. The scale exercised a protective action against decarburisation of the metal which nearly always occurred. The experiments indicate

that fuels rich in hydrogen will tend to cause rapid scale formation which cannot be stopped by reducing gases unless these are present in large proportions.

Influence of cold work and annealing on magnetic properties of thin wires of invar, iron, and steel. C. Biéler-Butticaz (Arch. Sci. Phys. Nat., 1927, **9**, 32—51).—In a previous paper (*ibid.*, 1925, **7**, 19) it was shown that for invar the internal friction increases suddenly at 340° in hard-drawn wire, and at 250° in annealed wire. These temperatures correspond to the maxima in the torsion moduli. It is now shown that no corresponding displacement occurs in the magnetic transformation point, although the form of the curve is altered by cold work. The magnetic susceptibility falls most rapidly between 200° and 250° both for cold-drawn and annealed wires. The magnetisation curve shows an anomaly at about 150°, which is attributed to the effect of cold work, and can be removed by annealing at 800°. A similar effect was observed in piano wire and in pure iron. A much smaller anomaly, attributed to the nickel transformation point, was observed at 340°.

C. J. Smithells.

Effect of prolonged annealing on the phosphide eutectic [in cast iron]. H. Pinsl (Stahl u. Eisen, 1927, **47**, 537—540).—After annealing at 980° for 48 hrs. a cast-iron rod containing 1·95% C, 2·46% Si, 0·30% Mn, 1·18% P, and 0·026% S, the microstructure of the cross-section showed the presence of three distinct zones, of which the central and outer consisted of practically pure ferrite saturated with phosphorus (0·8—1%), and containing coarse inclusions of graphite. Between these two zones was a ring of phosphide eutectic in globular masses surrounded by pearlite; the phosphorus content of this ring was as high as 1·98% in the middle, and decreased regularly towards the centre and edges of the bar. A sample of the metal taken from near the tuyères after a week's running of a furnace melting an iron containing 3·30% C, 2·20% Si, 0·40% Mn, 1·30% P, and 0·08% S, had the following composition:—0·03% C, 0·005% Si, 2·10% P, 0·12% S, 0·052% Cu, and Mn a trace, showing that the action of the hot air blast is to remove the carbon, silicon, and manganese, in preference to the phosphorus and sulphur. As this metal had been allowed to cool down very slowly with the furnace, its microstructure was entirely free of phosphide eutectic, and consisted of the saturated solid solution of phosphorus in iron containing inclusions of long needles of iron phosphide segregated along the grain boundaries.

A. R. Powell.

Oxygen in pig iron and cast iron. P. Oberhoffer and E. Pivovarsky (Stahl u. Eisen, 1927, **47**, 521—533).—Tables are given showing the variation in the composition, especially the oxygen content, of 20 pig irons from coke-fired furnaces, 14 pig irons from charcoal-fired furnaces, and 30 cast irons made up from many different types of raw material, full details of furnace dimensions, blast pressure and temperature, composition of feed to the furnace, and analyses of the slags produced being included. The mean hydrogen contents of the three varieties of iron examined were 13·5, 19·5, and 15·2 c.c./100 g., and the mean oxygen contents 0·022%, 0·023%, and 0·038% respectively. Practically the whole of the oxygen content in the second case was present as silica; in the other cases, however, there was no relation between the oxygen and silicon contents of the metal. The structure and properties of cast iron are seriously affected by the presence of more than 0·045% O. A high furnace temperature tends to reduce the proportion of included oxides in the castings, and this may be most satisfactorily obtained by increasing the quantity of air supplied; at the same time, this increases the rate of melting of metal, and consequently results in a greater output from the furnace. A. R. Powell.

Specific resistivity of copper-refining electrolytes and method of calculation. S. Skowronski and E. A. Reinoso (Trans. Amer. Electrochem. Soc., 1927, **51**, 113—131).—Sulphuric acid concentration is the main factor in obtaining electrolytes of low ohmic resistance, and the specific resistivity of sulphuric acid at different concentrations and at various temperatures has been determined. All resistivity measurements carried out at different temperatures are compared with measurements made at 55° (the normal operating temperature) as 100%, the comparison being called the "percentage resistivity." At constant temperature the specific resistivity of a solution of sulphuric acid containing 150 g./litre is taken as 100%, and any change in resistivity due to variation in the composition of the electrolyte is referred back to this standard figure. By using percentage resistivity data it is not only possible to calculate the resistivity from the chemical analysis of the electrolyte used, but also to make up any particular solution to any predetermined resistivity. The percentage resistivity of sulphuric acid decreases as the concentration increases, but is independent of the temperature within a wide range. Copper, nickel, and iron as sulphates increase the specific resistivity to a marked degree, whilst the effect of arsenic is slight; the increase is directly proportional to the amounts added. Glue has no effect on the resistivity of sulphuric acid solutions. Formulæ have been derived by means of which the resistivity of a tank-house electrolyte may be calculated from its chemical analysis.

M. E. Nottage.

Properties of electrolytic copper sheets. Sheets deposited on rotating cathodes. S. Sonoda (Trans. Amer. Electrochem. Soc., 1927, **51**, 191—205).—Electrolytic sheets produced on rotating cathodes are inferior in their physical properties to standard sheets rolled from cast ingots. This inferiority is connected with the microstructure; in ordinary annealed sheets the fine crystals are irregularly oriented, but in electrolytic sheets the crystals are developed perpendicular to the surface; hence the tensile strength is low in the direction perpendicular to the surfaces of the crystals. The ductility and malleability of electrolytic sheets is also low, and the sheets are difficult to work. The drawing, stamping, and compressive properties of the sheets were measured by the Erichsen test. Measurements were also made of the tensile strength and hardness. It was found that by rolling and annealing the electrolytic sheets, both surfaces could be made smooth, and the mechanical properties and microstructure developed were very

similar to those of ordinary copper sheets; annealing alone did not produce this effect. The direction of rolling had little effect, but heavy rolling produced marked physical changes. The best annealing temperature for the rolled sheets was 400—600°; annealing at temperatures higher than 800° gave rise to rapid grain growth and a decrease in the percentage elongation. Preliminary annealing before rolling is not necessary when the reduction is less than 50%. Electrolytic sheets rolled directly without annealing showed a tensile strength and percentage elongation greater than those for ordinary rolled copper sheets.

M. E. Nottage.

Gaseous reduction of tin concentrates. C. G. Fink and C. L. Mantell (Trans. Amer. Electrochem. Soc., 1927, **51**, 175—189).—The low-temperature reduction of stannic oxide by purified hydrogen is complete at 250°, the action being made irreversible by using an excess of hydrogen which sweeps out the water vapour formed; with stannic oxide in excess the action ceases as soon as the concentration of water vapour has reached N%, where N varies inversely as the temperature. The reduction takes place in one step or two, according as the temperature employed is above or below the thermal decomposition point of the lower oxide. The velocity of reduction is greatest at those points first affected; it is also dependent upon the velocity of diffusion of the water vapour formed. The reduced metal forms a fine grey powder with very little sintering even when the reduction takes place above the m.p. of tin. The temperature–time of reduction curves are logarithmic, taking the form $T_k{}^n M = C$, where T_k is the absolute temperature, M, the time in min., and n and C are constants; extrapolation of the curve shows that the reaction would be instantaneous at 1000°. The rate of reduction of ferric oxide by hydrogen has also been studied, the shape of the curve being the same as for tin, but the constants smaller; hence it is inferred that the reduction of ferric oxide by hydrogen takes place more easily than that of stannic oxide. The non-appearance of metallic iron in the reduced masses is probably due to the re-oxidation of the finely-divided metal. Temperature–time of reduction curves for Bolivian concentrates are of the same form as those for stannic oxide. The process of gaseous reduction depends for its success upon the absence of all slagging phenomena until reduction is complete, as even incipient fusion prevents the intimate contact of ore and gas. 80% of the tin metal content may be liquated from the non-fluid ore in this manner, the remaining 20% being almost completely recovered by fusing the residues at 1000—1050°, when the gangue forms a liquid slag from which metallic tin settles out. The economics of the hydrogen reduction of tin ores is also discussed. M. E. Nottage.

Diffusion law as applied to diffusion in solid solution. J. S. Dunn (J.S.C.I., 1927, **46**, 109 T).—A criticism of Tammann and Bredemeier (cf. A., 1925, ii, 541), who claim that the law of growth of films of reaction products upon metals is correlated with the mechanism of conduction of electricity in these films. Diffusion of gold in lead and of zinc in copper which should vary logarithmically with time upon their theory is shown to follow a parabolic law.

Patents.

Manufacture of steel. F. N. Speller (U.S.P. 1,617,726, 15.2.27. Appl., 16.8.26).—In the manufacture of steel, the ladle is preheated by pouring a quantity of superheated steel into it, prior to the addition of the bulk of the steel, which can then be held sufficiently long in the ladle to permit of separation of the non-metallic inclusion. T. S. Wheeler.

Plant for treating molten iron. E. C. R. Marks, From New Process Multi-castings Co. (E.P. 267,590, 15.12.25).—A blast furnace is used in conjunction with a number of electric furnaces mounted on a turn-table and arranged successively to receive molten metal from the blast furnace and to condition the metal as to composition and temperature. J. S. G. Thomas.

Annealing and hardening steel wires and bands. F. Köster (E.P. 254,278, 14.5.26. Conv., 23.6.25).—The wires etc., after passing through the annealing furnace, are drawn first through a pipe contained in a lead-bath and then through an oil-bath, air being excluded. M. E. Nottage.

Heat treatment of iron or steel. Automatic and Electric Furnaces, Ltd., and L. W. Wild (E.P. 267,253, 30.12.25).—In an electric furnace heated by direct current, magnetisation of the charge may be prevented by connecting a neutralising winding on the outside of the furnace in series with the heating winding, whereby a magnetising force equal and opposite to that generated by the heating winding is produced. Thus, magnetising coils carrying alternating currents, and detecting coils can be added to the furnace, and the change in the magnetic state of the charge from ferro- to para-magnetic can be detected. M. E. Nottage.

Case-hardening of steel articles. Rheinische Metallwaaren- u. Maschinenfabr. (E.P. 242,978 and 244,431, [A] 5.11.25, [B] 6.11.25. Conv., [A], 17.11.24, [B], 13.12.24.)—(A) After case-hardening by known methods, including annealing or subsequent heat-treatment, the article is subjected to a controlled heating agent, *e.g.*, a molten bath of lead, for such time that the carburised layer only is raised to the hardening temperature. The depth in the carburised layer to which "glass hardness" is to be produced is determined by trial. (B) When "glass hardness" is desired over the whole surface of steel articles the time of heating is maintained so that the heating to hardening temperature penetrates a short distance into the non-carburised core, whereby, on quenching, a normally hardened, uncarburised zone is formed between the carburised surface layer and the core. C. A. King.

Desulphurising fine iron pyrites. Metallbank u. Metallurgische Ges. A.-G. (E.P. 254,691, 7.6.26. Conv., 4.7.25).—The pyrites is deprived of part of its sulphur in a hearth or drum roasting furnace, and the remaining sulphur is driven out by aspirating or blowing air through the material in a Dwight–Lloyd blast roasting furnace, agglomerating the burnt pyrites if desired. The cooling between the operations may be with complete or partial exclusion of oxygen, whereby iron sulphate is formed. W. G. Carey.

Metallurgy of tin. E. A. C. Smith, Assr. to Guggenheim Bros. (U.S.P. 1,620,580, 8.3.27. Appl., 27.6.25).—

A partially reduced tin ore is leached with a solution of stannic chloride, ferrous chloride, and either hydrochloric or sulphuric acid, and the metallic tin, which is dissolved with formation of stannous chloride, is recovered by electrolysis, with regeneration of the leaching solution. A portion of this solution is electrolytically oxidised in a cell fitted with a diaphragm to form ferric chloride, and this solution is used to leach the tailings from the first leach, from which it extracts stannous sulphide, antimony, and bismuth. The latter metals are precipitated by treatment with metallic tin, and the solution of stannic chloride and ferrous chloride remaining is returned to the first leaching vessel.

T. S. Wheeler.

Preparation of [low-grade lead] ores for flotation. H. H. Hendrickson, Assr. to Grand Central Mining Co. (U.S.P. 1,620,761, 15.3.27. Appl., 9.9.26).—Low-grade lead ores are ground and mixed with sodium sulphide and water at about 70°. The product when submitted to flotation gives a 60% recovery of lead.

T. S. Wheeler.

Production of metallic [zinc] oxides. G. B. Filmer (U.S.P. 1,620,880, 15.3.27. Appl., 12.12.24).—Zinc is added to molten slag, which is heated in a furnace by passing an electric current through it. A current of air is mixed with the zinc vapour as it leaves the furnace. The slag removes any impurities in the zinc.

T. S. Wheeler.

Separation of zirconium and hafnium. D. Coster and G. von Hevesy, Assrs. to N. V. Philips' Gloeilampenfabr. (U.S.P. 1,618,960, 22.2.27. Appl., 13.3.24. Conv., 26.4.23).—A mineral containing zirconium and hafnium is fused with potassium hydrogen fluoride, and the mixture of potassium zirconium fluoride and potassium hafnium fluoride obtained is dissolved in a boiling aqueous solution containing 10% of hydrogen fluoride and 5% of potassium fluoride. The precipitate which separates on cooling contains less hafnium than the original material, and by repetition of the process a practically complete separation can be attained. To prepare metallic hafnium, a solution of the double fluoride is treated with an excess of a base, and the hydroxide precipitated is converted into the metal by any reaction suitable for the preparation of zirconium from its hydroxide.

T. S. Wheeler.

Metallurgical furnace. F. A. Fahrenwald (U.S.P. 1,623,469, 5.4.27. Appl., 12.10.25).—A number of one-piece hollow rolls are set horizontally and parallel in the furnace chamber, and have co-axial, solid shafts at each end projecting through the furnace walls. Provision is made for rotating all the rolls in the same direction, at the same speed.

W. G. Carey.

Heating metals by electricity. D. F. Campbell (E.P. 267,704, 3.3.26).—Materials to be heated, *e.g.*, wires or bars, are drawn through perforations in a block of magnetic material magnetised by a coil energised by an alternating electric current, the block being heated at about its temperature of recalescence by effects due to hysteresis and eddy currents.

J. S. G. Thomas.

Electrodeposition of chromium. C. Hambuechen, Assr. to Electro Metallurgical Co. (Re-issue 16,598, 19.4.27, of U.S.P. 1,544,451, 30.6.25).—See B., 1925, 726.

Refining brass and bronze secondary metals and their residues. T. Lewin (E.P. 269,253, 15.1.26).—See U.S.P. 1,574,043; B., 1926, 369.

Extrusion of metals [e.g., magnesium propellers]. E. Mitchell (E.P. 267,412, 23.9.26).

Method and apparatus for making metal wool from wire. A. K. Croad. From Brills Manuf. Co., Inc. (E.P. 268,244, 2.11.26).

Ore reduction (E.P. 266,402).—See I.

XI.—ELECTROTECHNICS.

Patents.

Electric furnace. A. D. Keene, Assr. to Westinghouse Electric and Manuf. Co. (U.S.P. 1,622,650—1, 29.3.27. Appl., [a, b] 24.4.25).—(a) A refractory resistor-supporting member has a portion projecting into the furnace, and is provided with a groove and locking means interfitting with the groove for holding it in the refractory wall of the furnace. (b) The resistor-supporting member is held in position in the furnace wall by means which are prevented from moving longitudinally.

J. S. G. Thomas.

Electric melting pot. F. Schuster (U.S.P. 1,623,209, 5.4.27. Appl., 16.12.25).—An electric heating element is arranged between an inner vessel and an outer jacket, in close proximity to the former. A heat-insulating ring is arranged between the upper edge of the inner vessel and an annular top plate secured to the upper edge of the jacket.

J. S. G. Thomas.

Electric battery. E. R. Gill (E.P. 266,565, 1.5.26).—A wet-battery system comprises a series of intercommunicating cells, having an inlet at one end and an outlet at the other end of the series. An arrangement of automatic valves permits the forward movement of fluid under pneumatic pressure from the inlet end of the series through the intermediate cells to the outlet end, where it is returned to the inlet end. A filling chamber is connected with the inlet, and is provided with a gas escape pipe having an automatic safety valve for controlling the pressure in the chamber.

J. S. G. Thomas.

Manufacture of storage batteries. C. M. Angell, Assr. to Vesta Battery Corp. (U.S.P. 1,622,304, 29.3.27. Appl., 5.11.23).—The surfaces of freshly-pasted battery-plates are dried, and the plates burned into groups in which they assume their final structural relationship of positive and negative plates, groups of which are assembled to form a battery.

J. S. G. Thomas.

Incandescence filament. G. R. Fonda, Assr. to General Electric Co. (U.S.P. 1,623,784, 5.4.27. Appl., 1.5.23).—An incandescence filament consists of tungsten and fractional percentages of thorium and copper.

J. S. G. Thomas.

Manufacture of articles from molten carbon. I. G. Farbenind. A.-G., Assees. of A.-G. für Anilin-Fabr. (E.P. 254,679, 18.2.26. Conv., 30.6.25).—Suitable relative movement is produced between a carbon electrode conveying sufficient current to fuse it, and a counter electrode, so that an article of desired shape—plate, rod, tube, etc.—is built up on the latter electrode by deposition of molten carbon from the electrode undergoing fusion.

J. S. G. Thomas.

Galvanic cell. H. D. NYBERG (U.S.P. 1,624,845 12.4.27. Appl., 24.5.22. Conv., 28.3.22).—See E.P, 195,580; B., 1923, 1184 A.

Production of physical and chemical changes in dielectric carbon compounds by means of ionised gases. H. E. R. VOGEL (U.S.P. 1,621,143, 15.3.27. Appl., 8.4.25. Conv., 26.3.20).—See E.P. 254,375; B., 1926, 757.

[Electrode-supporting sheets for] electric storage batteries or electric accumulators. FULLER'S UNITED ELECTRIC WORKS, LTD., and S. C. WHAITE (E.P. 266,497, 31.12.25).

Electric cell. G. WEISSMANN (E.P. 262,049, 20.8.26. Conv., 30.11.25).

Metallising insulating sheets etc. (E.P. 263,728).—See X.

XII.—FATS; OILS; WAXES.

Drying of fatty oils. A. V. BLOM (Z. angew. Chem., 1927, **40**, 146—147).—The behaviour of a thin film of a drying oil is dependent on the rate of formation therein of nuclei, consisting of molecular complexes. These have lower surface tension than the surrounding oil, and are consequently forced to the surface, where they pile together, their "solvate" sheaths being gradually compressed. Tackiness and yield value are the first evidence of this change, which ultimately leads to the formation of a surface skin. The "period of induction" is due to the slow rate of diffusion of these colloidal aggregates towards the surface. Chemical action does not begin until the concentration of micelles in the surface is sufficiently high to permit of molecular attraction. Solidification of the film then proceeds inwards in layers. The drying through of a film, *i.e.*, the production of a minimum of unchanged oil, thus depends on the relationship between thickness of layer and the accession of nuclei-inducing agents. By conversion to "stand oil," the formation of nuclei is inhibited, whilst some highly dispersed pigments, *e.g.*, French ochre, carbon blacks, etc., adsorb nuclei already formed. On the contrary, a tendency to assist nucleus formation constitutes the drying action of metallic soaps and certain pigments. In poppy seed and similar oil-films, the surface micelles are more strongly "solvated" than in linseed oil, and the film has too loose a structure for condensation reactions to occur, whilst tung oil, on the other hand, has more surface activity. Gelatinisation is not a normal stage in the drying of an oil film, but may occur when too many nuclei are formed, as in air blowing or admixture with stand oil. Feeble drying power is a consequence of this anomalous gelatinisation. S. S. WOOLF.

Chemistry of palm oil. A. RAYNER (J.S.C.I., 1927, **46**, 160 T). W. BRASH (*ibid.*, 160 T).—Polemical.

Saturation of resin acids. MARGOSCHES and others. —See XIII.

Determination of fat in milk. POPP.—See XIX.

Glycerol tables. BOSART and SNODDY.—See XX.

PATENT.

Treatment of oils (E.P. 267,174).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Arsenic in printing ink. T. H. BARRY (Analyst, 1927, **52**, 217—221).—Suggested limits for arsenic in printing ink pigments which are to be used on confectionery wrappers are (1) pigments which may be used unconditionally, containing less than 1 pt. of arsenic in 50,000 of pigment; (2) those containing between 1 in 20,000 and 1 in 50,000, to be used in conjunction with those in class (1); and (3) those containing over 1 pt. in 20,000, to be rejected. The ink contains also considerable proportions of oil, and a maximum of less than 1 pt. of arsenic in 50,000 of ink is assured by the above limits, but this method of fixing limits is unsatisfactory, and one in terms of the weight of arsenic per unit area of wrapper or carton is suggested. D. G. HEWER.

Degree of saturation of the resin acids. II. B. M. MARGOSCHES, K. FUCHS, and W. RUZICZKA (Chem. Umschau, 1927, **34**, 73—76).—In continuation of earlier researches (B., 1927, 228), it is pointed out that in the determination of iodine values by the rapid method (*ibid.*) the halogen acids produced correspond to 50% of the iodine consumed, whilst in the case of resins or resin acids the amount may rise to 80—90%. From the examination of numerous mixtures of oleic acid and various resins, it is demonstrated that an empirical value, the "Plussäure," which is the excess of halogen acids produced over 50%, bears a linear relation, within limits, to the amount of resin in the mixture. Hence, multiplication of this value by a factor, in this case 1·4, gives an approximate value for the amounts of resin in such mixtures. With mixtures containing more than 30% of resins the relation does not hold, the "Plussäure" value increasing less rapidly than the amount of resin. In practice the iodine value of a mixture is determined by the Margosches rapid method (*ibid.*), the excess of halogen back-titrated directly, then 20 c.c. of a 3% solution of potassium iodate are added and the halogen liberated is determined by a further titration. Although indirect and approximate only, the method is claimed as rapid, and of especial value in technical soap analysis. E. HOLMES.

PATENTS.

Manufacture of compositions containing colouring materials [pigments etc.]. W. H. WHATMOUGH (E.P. 267,207, 7 and 22.12.25).—Insoluble pigments or lakes are precipitated in the presence of dilute aqueous dispersions of oleaginous materials (*e.g.*, drying oils, mineral oils, waxes, etc.), the latter separating from the aqueous phase and being carried down by the pigment. The composite masses so produced (the consistency of which is controllable) are characterised by ready dispersion, and are used in printing inks, paints, typewriter ribbons, etc. S. S. WOOLF.

Coating wood, iron, or other structures. W. BAUR (E.P. 252,364, 14.5.26. Conv., 20.5.25).—Stationary structures, particularly of iron or wood, are coated with a refractory varnish which is "burnt on" at 50—600° by means of a hot-air blast. A suitable coating is prepared by heating castor oil at 280—300° with 5—10% of metal oxide (lead, cobalt, zinc, etc.) until the mixture is clear. On cooling, 10—20 pts. of this wax-like material are incorporated with 80 pts. of an

ester gum–linseed or tung oil varnish base and thinners. To 80 pts. of this medium are added 20 pts. of metal powder, *e.g.*, aluminium, brass, or lead. S. S. WOOLF.

Manufacture of lead compounds. R. WILHELM, Assr. to COMMONWEALTH WHITE LEAD & PAINTS PROPRIETARY LTD. (U.S.P. 1,617,887, 15.2.27. Appl., 24.9.25. Conv., 30.3.25).—See E.P. 249,809; B., 1927, 259.

Manufacture of resins. C. O. TERWILLIGER, Assr. to F. VON BRIESCU (U.S.P. 1,624,082 and 1,624,638, 12.4.27. Appl., [A] 29.6.23, [B] 5.7.23. Renewed [A, B], 7.2.27).—See E.P. 218,277 and 218,638; B., 1926, 890, 999.

Production of fibres etc. (F.P. 612,879).—See V.

Cellulose composition (U.S.P. 1,620,977).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Coagulation phenomena in *Hevea* latex. IV. Latex in the second liquid zone. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Comm. Netherlands Rubber Testing Stat., Buitenzorg, 1927, No. 6, 503—565. Arch. Rubbercultuur, 1927, **10**, [11]; cf. B., 1925, 17, 891).—Latex to which an excess of acid has been added attains a second zone of stability in which the sign of the electric charge on the globules has changed from negative to positive. The coalase in such latex is killed, but the latex gradually "creams" and the top layers ultimately coagulate. The latex coagula are paler in colour than the earlier ones, and contain a lower proportion of nitrogen. The rubber is always low in viscosity and very plastic, but has a tendency to develop tackiness, especially if exposed to light; in extreme cases it finally becomes hard and resinous. D. F. TWISS.

Ball rubber (rubber prepared after the Brazilian method). O. DE VRIES and W. SPOON (Comm. Netherlands Rubber Testing Stat., Buitenzorg, 1927, No. 8, 1—65; Arch. Rubbercultuur, 1927, **10**, [1]).—The Brazilian method for the preparation of rubber from latex has been adopted for a time in Java on a large experimental scale; the method, however, showed no especial advantages, and the normal procedure for the production of plantation rubber has now displaced it. In its properties generally ball rubber appeared to be intermediate between ordinary plantation rubber and and hard fine Parà rubber. D. F. TWISS.

Direct determination of hydrocarbon in raw rubber, gutta-percha, and related substances. A. R. KEMP (Ind. Eng., Chem., 1927, **19**, 531—533).—The hydrocarbon of rubber or of gutta-percha can be determined by a modification of the Wijs method. A 0·1 g. sample, from which the resins have been removed by any suitable method, is swollen overnight in 75 c.c. of purified carbon disulphide; 25 c.c. of 0·2*N*-Wijs solution are added with agitation so as to obtain a clear red solution. The flask stoppers are wetted with a drop of potassium iodide to prevent escape of iodine, and the flasks are kept in ice water for 2 hrs.; 25 c.c. of 15% potassium iodide solution and 50 c.c. of distilled water are then introduced, and the iodine is titrated with 0·1*N*-sodium thiosulphate. The theoretical iodine value for C_5H_8 with one double linking is 372·8. It is unnecessary to exclude diffused light, and a large excess of the Wijs reagent does not affect the results. The results for samples of extracted rubber, gutta-percha, and balata correspond closely with the theoretical values, any discrepancy being in accord with the known protein, dirt, and humus contents. Milling for 2 hrs. in air or melting by prolonged heating in carbon dioxide at 280° causes no appreciable change in the chemical unsaturation of rubber. D. F. TWISS.

Cloth dyeing for rubber proofing. HOCKNEY and BANCROFT.—See VI.

PATENTS.

Preparing rubber compositions. G. W. ACHESON (U.S.P. 1,623,517, 5.4.27. Appl., 21.4.23).—Rubber latex is mixed with aqueous suspensions of deflocculated fillers and sulphur; coagulation of the rubber and flocculation of the filler are effected simultaneously with formation of a precipitate containing the ingredients in intimate admixture. D. F. TWISS.

Removal of liquid polymerides from synthetic rubber. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 267,808, 3.9.26).—The rubber-like masses obtained in the polymerisation of butadiene are accompanied by liquid polymerisation products. These are removed by applying active charcoal to the sheeted product, heating if desired at 40—50° for several hours under reduced pressure, and then removing the charcoal mechanically or by washing with water. D. F. TWISS.

Process and apparatus for devulcanising rubber. E. E. ROYER (F.P. 611,405, 30.5.25).—Old rubber is heated with a liquid such as mineral oil in an autoclave, from the bottom of which the resulting solution is forced into a still. The mineral oil which is recovered by distillation is returned to the autoclave, whilst the residual "reclaimed" rubber is removed from the still at the end of each distillation process. D. F. TWISS.

Preservation of latex. A. E. JURY and O. H. SMITH, Assrs. to GENERAL RUBBER CO. (U.S.P. 1,619,938, 8.3.27. Appl., 8.6.26).—Latex is treated with an organic antiseptic and an alkaline phosphate.

T. S. WHEELER.

Apparatus for vulcanising under fluid pressure. R. D. FRITZ, Assr. to B. F. GOODRICH CO. (U.S.P. 1,622,438, 29.3.27. Appl., 12.1.26).

Coating materials with rubber (E.P. 261,700).—See XI.

XV.—LEATHER; GLUE.

Fluorescence test for synthetic and natural tannins. O. GERNGROSS and G. SANDOR (Collegium, 1927, 12—20).—Solutions of a number of synthetic tannins (1 : 1000) show violet or bright blue fluorescence in the light of a quartz lamp. Some synthetic tannins (*e.g.*, Maxyntan) show no fluorescence. 1 : 1000 solutions of vegetable tanning materials do not give characteristic fluorescence except oakwood extract, which shows green, donga reddish, pine bark and malet bluish-violet, and quebracho and tizerah yellowish-green. Acidification destroys the fluorescence, alkali changes the donga fluorescence to orange, and that of pine bark and malet to grass-green, and destroys that of quebracho and tizerah. Fibres treated with quebracho, tizerah, and mimosa show a yellow fluorescence, and to a smaller

extent when treated with other vegetable tannins. Pine bark, malet bark, larch bark, hemlock and chestnut extracts all give a violet fluorescence on cellulose fibres treated with them. The treated fibre soon loses its fluorescing property unless a small amount of sublimate is added to the solution. The violet fluorescence is probably caused by some substance akin to fisetin.

D. Woodroffe.

Adsorption of the violet fluorescing substances from pine bark extract and the yellow fluorescing substances from quebracho wood by different adsorbents. Relation between adsorption and chemical constitution. O. Gerngross, G. Sandor, and K. Tsou (Collegium, 1927, 21—24).—1 : 500 solutions of pine bark and quebracho extracts, respectively, were treated with different acetyl- and nitro-cellulose fibres. Different nitrocelluloses absorbed the fluorescing material from quebracho solutions, but not from pine bark. Cellulose diacetate, "cellit" powder, and acetate-silk alone absorbed the fluorescing agent in pine bark solutions. Hydrocellulose, starch, agar-agar, wool, silk, and casein absorbed the fluorescent agent from both solutions. The irreversible adsorption diminished with increasing p_H value.

D. Woodroffe.

Detection of quebracho tannin and similar tannins in extract mixtures and in leather. L. Pollak and W. Springer (Collegium, 1927, 46—52).—1 g. of solid or dried extract is mixed into 10 g. of caustic soda solution (d 1·32) in a silver crucible, slowly heated to 260° with stirring until the dried mass re-melts, poured into hot water, neutralised with sulphuric acid (1 : 1), made up to 500 c.c., and 100 c.c. are shaken out into three lots of 50 c.c. of ethyl acetate, the ester extracts dehydrated over anhydrous sodium sulphate, the ester distilled off, and the residue weighed, and calculated per 100 pts. of dried extract. A similar determination is made on the precipitate obtained in the formaldehyde-hydrochloric acid test, using 100 c.c. of tannin solution. It is proposed to use such figures for detecting and identifying different tannins. Tabulated results are given, together with the fluorescence of solutions of the different extracts, of the residue from the ester extracts of their fused mass, and of the fused mass of the formaldehyde precipitate, also the colour of the ester after shaking with the solution for the fluorescence reaction. These tests provide a means of distinguishing the sub-group quebracho, tizerah, urunday, and mimosa bark from the larger group of pyrocatechol tans, even in mixtures and when strongly sulphited. For leathers, the formaldehyde precipitate from the water-soluble matter can be used, or the leather can be fused with caustic soda and treated as above.

D. Woodroffe.

Waste hide as manure. Joret and Radet.—See XVI.

Patents.

Manufacture of condensation products containing sulphur [tanning agents]. I. G. Farbenind. A.-G., Assees. of A.-G. für Anilin-Fabr. (E.P. 252,694, 27.4.26. Conv., 28.5.25. Addn. to E.P. 218,316 ; B., 1925, 181).—Products having properties similar to those previously described, except that they are only slightly hygroscopic, are prepared by the interaction of hydroxyarylcarboxylic acid sulphochlorides, such as salicylic acid sulphochloride, with esters derived from hydroxyaryl-compounds, such as 2-chlorophenyl carbonate, resorcinyl diacetate, or acetylsalicylic acid.

L. A. Coles.

Process of tanning. J. K. Tullis (U.S.P. 1,621,612, 22.3.27. Appl., 3.10.23).—Hides are partially tanned in weak tanning liquors, dried till bone dry, and then drummed with half their weight of strong tanning liquor to complete the tannage.

D. Woodroffe.

Leather dressing and tanning material. F. C. Atkinson (U.S.P. 1,622,127, 22.3.27. Appl., 16.8.19). —Corn cobs are digested under pressure at 140—200° for 2—3 hrs.

D. Woodroffe.

Manufacture of glues, gelatins, and the like from plagiostomaceous materials. A. Ehrenreich (E.P. 267,041, 4.11.26. Conv., 12.7.26).—Skins, pectoral and other fins, damaged skins and all gelatinous waste of plagiostomi are washed in water, or limed for 2 weeks, washed and bleached with sulphurous acid, then treated with water in a dissolving vat provided with a perforated partition, composed of a grid of wooden laths or galvanised wire cloth, and a heating coil. The liquid is heated until it froths or bubbles (70—80°), the heating discontinued and re-started alternately for 2 hrs. or until the liquid has reached the desired consistency, the impurities are removed, the viscous liquid is transferred to a receptacle which is placed in a refrigerator, or mixed with 30 g. of boric acid per 100 litres of liquor, and allowed to set in hermetically sealed flat boxes to obtain sheets of gelatin or poured into a vat, cooled to 40°, then 2½ kg. of hydrochloric acid or 28% acetic acid are added per 100 litres of liquor, and finally oil of sassafras to produce a liquid glue.

D. Woodroffe.

Artificial leather (F.P. 611,401).—See V.

Treatment of yeasts (E.P. 243,373).—See XVIII.

XVI.—AGRICULTURE.

Value of "dissolved" waste hide as a manure. G. Joret and E. Radet (Ann. Falsif., 1927, 20, 133—148).—In the solution process, which consists in boiling the hide with sulphuric acid and adding calcium phosphate to increase the consistency and partially neutralise the acid, a variable proportion of nitrogen is rendered soluble in water, depending on the amounts of acid and phosphate used. In all cases there is an insoluble residue, which is useless for agricultural purposes. When mixed with a highly calcareous earth, the mean value of the nitrifiable nitrogen may amount to two thirds, but with a non-calcareous earth to only one half of that for the same amount of desiccated blood. Free acid and tannin tend to hinder nitrification. The Street index is of no value in evaluating these products, but the determination of the insoluble residue, its nitrogen content, as well as the intensity of the tannin reaction, are useful indications as to their commercial value as manures.

F. R. Ennos.

Nitrification of ammonium sulphate in soil. H. J. Harper and B. Boatman (J. Amer. Soc. Agron., 1926, 18, 876—888).—Air-drying soils does not appear to affect the nitrification of ammonium sulphate. A

higher percentage of nitrate-nitrogen was produced when smaller amounts of ammonium sulphate were used. Phosphorus and potassium fertilisation did not increase the nitrifying power of soils better than ground limestone. CHEMICAL ABSTRACTS.

Biological activities in fertiliser composts. A. J. ENGLEHORN (Proc. Iowa Acad. Sci., 1925, **32**, 85—94).—The presence of sulphur in a fertiliser compost reduces, and that of orthoclase or raw rock phosphate increases, the number of bacteria present; the presence of sulphur stimulates, and that of orthoclase depresses, the growth of fungi. CHEMICAL ABSTRACTS.

Determination of the fertiliser requirements of soils by the Neubauer seedling method. S. GERICKE (Z. angew. Chem., 1927, **40**, 366—368).—100 rye seedlings were grown for 18 days in 100 g. of the soil to be tested in glass bowls. The young plants were then washed free from soil and their potash and phosphorus contents determined. If these fall below certain values, originally given as 8 mg. P_2O_5 and 24 mg. K_2O, the soil is considered to be deficient in the fertiliser concerned. The method is not applicable to nitrogen. Various critical investigations of this method are discussed, and it is concluded (1) that the period of growth should be 14—15 days, in which time a maximum absorption of the fertiliser is obtained; (2) the soil should be diluted with sand and a blank test performed to compensate for interchanges between the plant and the sand, of minerals originally present in the seed; (3) the amount of water added, temperature, and light intensity all have a slight effect on the results, and should be standardised. The amount of fertiliser found in the grown seedlings which is considered to represent a satisfactory soil also varies with the species of plant grown. In spite of these limitations, the method gives results much more closely in accordance with field trials than are the indications of soil analysis by chemical methods, and is of great value. C. IRWIN.

Fungicidal and bactericidal action of selenium and tellurium compounds. N. M. STOVER and B. S. HOPKINS (Ind. Eng. Chem., 1927, **19**, 510—513).—Sodium selenite and tellurite, potassium selenite and tellurite, and tellurium hydrogen tartrate in 0·05—0·0005*N*-solutions have practically no fungicidal action on the fungus causing chestnut blight, *Endothia parasitica*, even after exposure for a week, whilst selenious acid, lithium and thallous selenites are definitely toxic. Apple blotch, *Benturia inæqualis*, is not controlled by 0·1—0·04*N*-selenious acid even after repeated application. Selenious acid and thallous selenite are quite bactericidal to pear blight, *B. amylovorus*, according to laboratory tests, but these results have not been substantiated in the field. Selenious acid injected into pear and chestnut trees enters the circulatory system, but appears to be too injurious to the tree to be usefully employed against parasitic fungi. On the other hand, it is sufficiently toxic to be used as a herbicide against dandelion, Canada thistle, and burdock. E. HOLMES.

Ionic exchange of zeolitic silicates. KAPPEN and RUNG.—See VII.

Fumigation tests with chloropicrin. HOYT and ELLENBERGER.—See XIX.

PATENTS.

[Seed] disinfecting composition. Non-hygroscopic seed disinfectants. M. ENGELMANN and A. R. ALBRIGHT, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,618,369 and 1,618,370, 22.2.27. Appl., [A], 11.1.23; [B], 8.6.23).—(A) An insoluble, organic mercury compound, *e.g.*, *o*-acetoxymercuriphenol (10—30%), is intimately mixed with a dry, pulverised, alkaline-earth oxide or hydroxide (90—70%). (B) A mercuriphenol is mixed with an alkaline carbonate and an alkaline-earth oxide or hydroxide. T. S. WHEELER.

Production of [seed] disinfectants. M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,618,371—2, 22.2.27. Appl., [A], 19.2.24; [B], 25.7.25).—(A) Finely-divided calcium carbonate or other insoluble basic substance (1000 pts.), copper sulphate or other soluble copper salt (500 pts.), and water (6000 pts.) are agitated together, and the product is filtered off, dried, and ground. (B) Mercuric acetate (50 pts.), kaolin (500 pts.), sodium *o*-nitrophenoxide or similar compound (23 pts.), and water (1500 pts.) are heated at 80° for 4 hrs., when the corresponding mercuriphenol compound is obtained, dispersed on the kaolin; its value as a seed disinfectant is thus increased. T. S. WHEELER.

Lactonitrile fumigant. L. J. CHRISTMANN, Assr. to AMERICAN CYANAMID Co. (U.S.P. 1,620,074, 8.3.27. Appl., 3.8.25).—An insecticide comprises a mixture of a stable organic cyanogen compound, *e.g.*, lactonitrile, (50 pts.), a non-alkaline saponaceous adhesive material, *e.g.*, saponin, (25 pts.), and water (50 pts.). It is further diluted with water before spraying. T. S. WHEELER.

Preparation of a [sulphur] fungicide. J. H. REEDY and W. S. BROCK (U.S.P. 1,620,229, 8.3.27. Appl., 3.10.24).—Calcium polysulphide solution is treated with zinc sulphate, and the mixture of calcium sulphate, zinc sulphide, and finely-divided sulphur obtained is employed as a fungicide. T. S. WHEELER.

Dehydrating vegetable substances etc. (E.P. 267,203).—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Wort boiling. L. R. SKINNER (J. Inst. Brew., 1927, **33**, 219—222).—The crystalline structure of copper, which is universally chosen as the container in which to boil wort, explains its exceptional heat-conductive power. Heat radiation is an ionic emission, and negatively charged ions pass from the source of heat—the furnace—to the copper. In the cases where the copper has been "gassed" by the reducing action of the flue gases on the oxygen contained in the copper, the cohesion between adjacent crystals is reduced, and a quicker passage is offered to the ions of emission, the rate and velocity of impingement of which may have a direct bearing on the ultimate cooked flavour of the wort. The cooking value of steam-heated coppers is inferior to that of fire-heated coppers, since the energy is third-hand, and there is not sufficient difference of temperature between the steam and wort to get the true cooking effect and complete sterilisation. With pressure steam-heated coppers, owing to the higher temperatures, sterilisation is better, the concentration is greater, and the rate of evaporation is increased with an improved cooking

effect. Steam, and even super-steam heated pressure coppers do not compare favourably with fired coppers, which ensure a better quality of heat and a higher heating temperature. Coal and oil give the sun's energy second-hand, whereas energy transmitted by steam is third-hand, owing to the coal or oil having to be burned to generate the steam. C. Ranken.

Production of *n*-butyl alcohol and acetone from Jerusalem artichokes. A. C. Thaysen and B. M. Green (J. Inst. Brew., 1927, **33**, 236—240).—The tubers of Jerusalem artichokes (*Helianthus tuberosus*) cannot be converted into *n*-butyl alcohol and acetone by *Bac. butylicus*, Weizmann, in the same way as raw materials containing starch, owing to the bulk of the carbohydrates in the tubers not being directly fermentable by the organism. A yield of 12 gals. of " oil," or more, per ton of fresh tubers can, however, be obtained if the original carbohydrates are hydrolysed and the mashes diluted to such an extent that the reducing sugars are prevented from exerting their inhibitory action on the micro-organisms or their enzymes. C. Ranken.

Difficulties of the sulphuric acid treatment [of distillery mashes]. Wendel (Z. Spiritusind., 1927, **50**, 96—97).—If lactic acid is replaced by sulphuric acid in the treatment of distillery mashes, owing to the greater strength of the latter acid, measurement of the resulting acidity to litmus by titration is insufficient, and must be amplified by determinations of the hydrogen-ion concentration. The efficiency of the treatment depends not only on the strength of the acid, but also on the concentration of the mash and the kind of raw material. Mashes made with maize require a lower degree of acidification than those made with potatoes or rye, whilst, in addition, greater amounts of acid can be safely added to thick mashes. If ammonium sulphate is used as a nitrogenous pabulum for the yeast, regard must be had to the increase of acidity from this cause during fermentation. Satisfactory results are obtained with sulphuric acid with mash concentrations varying from 15—18° Balling, and with p_H 5·0 (approx.). For the chief mash, the optimal acidity to litmus is given as 0·15—0·20°, and as 0·3° or slightly higher for the subsidiary mash. C. Ranken.

Patents.

Treatment of yeasts by autolysis. M. Kahn, E. le Breton, and G. Schaeffer (E.P. 243,373, 19.11.25. Conv., 19.11.24).—Food products and products for tanning and tawing are obtained by autolysing distillery yeast, rhizopus, and amylomyces. This is effected in a medium of 5—20% by wt. of sodium chloride, for a time varying from a few hours to 6 days, the temperature being maintained between 40° and that at which the ferments are destroyed. The autolysis may be accelerated by adding suddenly after a few hours' action 5—10% of sodium chloride to the mass. (Cf. E.P. 225,228 ; B., 1925, 605.) A. Rayner.

Manufacture of beverages. H. Mackeson (E.P. 267,236, 14.12.25).—The juice or must of fruits, particularly apples, pears, or grapes, is mixed with malt extract, fermented, and filtered. It is then further mixed with a priming solution consisting of glucose syrup or a concentrated fruit juice, with the addition of malt extract, again filtered, and pasteurised. F. R. Ennos.

Production of lactic acid from sugar-containing raw materials by means of lactic acid bacteria. A. Pollak (U.S.P. 1,624,674, 12.4.27. Appl., 11.9.24. Conv., 29.9.23).—See E.P. 222,514 ; B., 1926, 73.

Manufacture of butyric acid and other aliphatic acids. L. Le Franc, Assr. to Lefranc & Cie. (U.S.P. 1,625,732, 19.4.27. Appl., 18.8.22. Conv., 26.9.21).—See E.P. 186,572 ; B., 1923, 674 a.

XIX.—FOODS.

Determination of fat in milk by the Neusal method. M. Popp (Chem.-Ztg., 1927, **51**, 270).—Sufficient solution for 10,000 determinations is made by dissolving 10·2 kg. of a mixture of sodium salicylate and citrate in 40 litres of water. After remaining overnight a slightly insufficient quantity of butyl alcohol (9800—9850 c.c.) is added, and determinations of fat with this solution on a series of milks are compared with those made by the sulphuric acid method. The amount of the alcohol is then adjusted so that the results obtained by the two methods are in agreement. For the fat determination, 4 c.c. of the above solution are mixed with 9·2 c.c. of milk in a butyrometer, heated at 65°, centrifuged for 5 min., and the fat read at 45°. F. R. Ennos.

Immersion refractometer and its value in milk analysis. G. D. Elsdon and J. R. Stubbs (Analyst, 1927, **52**, 193—214).—Results from comprehensive tests with the immersion refractometer as a means of detecting added water in milk show that claims made in this connexion have been much exaggerated. Refraction is found to be proportional to the solids-not-fat, and in no case did a milk having a refraction over 37·1 have less than 8·5% of solids-not-fat. Also since refraction rises as acidity increases, up to a point, a watered milk may on keeping give a figure identical with that of the genuine milk. It is difficult to apply a correction factor for such increase due to acidity, but generally, on an average, an increase of 1·0 in the acidity (c.c. of 0·1*N*-sodium hydroxide required to neutralise 10 c.c. of milk) accompanies an increase of 0·2 in the refraction. D. G. Hewer.

Basic viscosity of ice-cream mixes. A. Leighton and O. E. Williams (J. Physical Chem., 1927, **31**, 596—600).—Ice-cream mixes show a " basic " viscosity, *i.e.*, a viscosity independent of the changes in mechanical structure which occur on being kept. At constant temperature the basic viscosity changes with the concentration of water in accordance with the Arrhenius equation, $\log \eta = \theta c$. L. S. Theobald.

Formaldehyde in fish. D. B. Dill and P. B. Clark (Analyst, 1927, **52**, 222 ; cf. B., 1926, 339).—Formaldehyde obtained from the fish examined by the authors is regarded as not due to oxidation of trimethylamine (cf. Tankard and Bagnall ; B., 1927, 25) since little or no formaldehyde was present at canning time, but developed with age in the absence of oxygen, and tests were made on distillates obtained by steam

distilling the samples mixed with a 2% solution of phosphoric acid (c.c./g.), the volume being kept constant by immersing the flask in a heated salt bath.

D. G. HEWER.

Quality of the protein of whale meat products. W. L. DAVIES (J.S.C.I., 1927, **46**, 99—100 T).—Attention is drawn to the high content of non-protein nitrogen in feeding stuffs manufactured from animal by-products and refuse. This non-protein nitrogen, which is water-soluble, has been examined in the case of whale meat flakes and whale meat meal, comparing the composition of the aqueous extracts in each case with the composition of the pure tissue protein. With respect to diamino-acid content, the aqueous extracts compare favourably in composition with pure tissue protein, and also with any complete protein, and, although the total nitrogen allotted to diamino-acid is slightly lower for the extract, this is counterbalanced by a higher amount of nitrogen allotted to monoamino-acid nitrogen in the extract. Such extracts are slightly inferior in composition to similar extracts of various fish meals. The flake had roughly a fifth, and the meal two fifths, of its nitrogen as non-protein water-soluble nitrogen. The flakes kept better under ordinary conditions of storage than the meal.

Changes during storage in the composition of mangels. J. P. DREW and G. T. PYNE (J. Dep. Lands Agric., Ireland, 1926, **26**, 9—14).—The composition of fresh Red and Yellow Intermediate varieties, respectively, was: dry matter 9·43, 11·21; sucrose 4·55, 6·48; reducing sugars 0·16, 0·22; crude protein 0·89, 1·14; true protein 0·39, 0·55; oil 0·03, 0·03; fibre 0·65, 0·76; ash 1·13, 1·13%. On storage for 5 months the percentages of oil, fibre, ash, and true protein were scarcely changed; both varieties lost 28% of the dry matter and 26—40% of the sucrose. The crude protein decreased 0·17—0·35%; the amounts of nitrate nitrogen decreased from 0·041 to 0·025, and 0·045 to 0·026%, respectively. CHEMICAL ABSTRACTS.

Examination of foodstuffs for preservatives. A. C. CHAPMAN (Analyst, 1927, **52**, 215—217).—From certain carbohydrate materials such as caramel an acid, or mixture of acids, may be obtained which closely simulates benzoic acid. It gives a violet coloration with ferric chloride, apparently due to a phenolic substance present in smaller proportion than the acid. The latter reacts with bromine, but gives no precipitate. Formaldehyde, or some substance very closely resembling it, is produced on heating sugar products, and formaldehyde reactions in such cases do not necessarily indicate addition of that substance. Boron occurs naturally in agar-agar and other seaweeds. D. G. HEWER.

Fumigation tests with chloropicrin [on food-stuffs etc.]. L. F. HOYT and E. P. ELLENBERGER (Ind. Eng. Chem., 1927, **19**, 461—464).—A large variety of food products, fumigated on the commercial scale with nitrochloroform for a week, at concentrations of 0·8—1·33 lb./1000 cub. ft. of storage space, appear to be undamaged in any way. Germination tests on corn, oats, sunflower, wheat, and buckwheat show that exposure to chloropicrin at the lower concentration for one week had no deleterious effect on the germination of the seed, but rather the reverse effect on the last two. Chloropicrin is recommended as a fumigant on the grounds of the protective warning afforded by its lachrymatory power in high dilutions, and because of its highly poisonous effect on such insects as the Indian meal moth, Mediterranean flour moth, and the more resistant confused flour beetle. E. HOLMES.

PATENTS.

Manufacture of bread. J. MATTI (E.P. 266,967, 25.6.26).—100 pts. of flour, 66·5 pts. of water, 2 pts. of salt, and 1 pt. of yeast are mixed at 38—40°, 0·1 pt. of an edible neutral fatty substance, *e.g.*, refined paraffin oil, is added, the whole mechanically kneaded for 12 min., and fermented for 40 min. After the moulding and rising in the pan, which occupy 15—30 min., the mixture is baked for 1 hr. F. R. ENNOS.

Manufacture of bread. H. A. KOHMAN (E.P. 244,489, 14.12.25. Conv., 12.12.24).—The usual ingredients of the dough are mixed with a dough-maturing agent, such as potassium persulphate, bromate, iodate, or periodate, in a quantity greatly in excess of that permissible when the dough is kept for the ordinary fermentation period. After mechanically mixing the batch for 10—20 min., it is divided, proofed, and baked in the usual way. F. R. ENNOS.

Milling products [flour]. H. C. J. H. GELISSEN, Assr. to NOYADEL PROCESS CORP. (U.S.P. 1,620,458, 8.3.27. Appl., 3.5.26. Conv., 11.3.25. Cf. E.P. 102,967; B., 1918, 71 A).—Flour is treated with a current of air at 20° in presence of a small quantity (0·003%) of an aldehyde, *e.g.*, acetaldehyde or benzaldehyde, and an acid anhydride, *e.g.*, acetic or succinic anhydride, together with a catalyst, *e.g.*, cobalt acetate or a similar salt of vanadium, chromium, etc. The peroxide produced bleaches the flour. T. S. WHEELER.

Butter and cream treating process. R. K. COONEY, Assr. to CAMPBELL-COONEY PATENTS CO. (U.S.P. 1,599,649, 14.9.26. Appl., 30.12.24. Renewed 24.2.26).—Highly acid cream is agitated with a small quantity of slaked lime at 38°, pasteurised, and centrifuged to remove solid matter. The keeping properties of the cream, and of the butter prepared from it, are improved.

T. S. WHEELER.

Pickling of fruit etc. J. W. BECKMAN, Assr. to G. C. ROEDING (U.S.P. 1,621,188, 15.3.27. Appl., 8.12.25).—The fruit, such as olives, is treated with a 2% solution of caustic soda for 4—6 hrs. to open the pores, and then with 1·5% hydrochloric acid and brine solution for 2—4 days. It is then immersed in a 1% solution of sodium lactate and lactic acid, the time required to remove the free hydrochloric acid and give the fruit the desired acidity being approximately one week.

F. R. ENNOS.

Preserving fruit juices and other liquids. W. MATZKA (E.P. 267,058, 5.9.25).—The liquid passes slowly through a vessel where it is subjected to gentle electrolysis between a gold cathode and an aluminium anode. The temperature of the liquid, which is heated by passing steam or hot water through the electrodes, is regulated between 30° and 55°, depending on its albumin content and

acidity, and is substantially below the normal pasteurisation temperature, so that the natural aroma is retained.

F. R. ENNOS.

Dehydrating vegetable substances or products of organic character. B. J. OWEN (E.P. 267,203, 7.12.25).—The drying agent, consisting of heated air, is supplied to the mass of material under treatment (*e.g.*, grain or root-crops) at ranges of temperature, pressure, and volume such as have been found to regulate the consolidation of the mass satisfactorily, and also to promote the naturally occurring physical and chemical reactions, so that the rate of dehydration is increased to a maximum, and the effects of the exothermic reactions occurring are utilised to the best advantage.

F. R. ENNOS.

Preparation of vitamin-rich products. H. ISCOVESCO and A. B. ADAMS (E.P. 267,410, 17.9.26).—Cod liver residues are treated with a solution of caustic soda (d 1·33) and kept at about 18° for 2 months or longer. The saponified mass, after drying *in vacuo* below 50°, is extracted with acetone; the solvent is then evaporated, and the vitamin-rich product dissolved in liquid fats or glycerin before addition to foodstuffs.

F. R. ENNOS.

Production of fish-oil powder. W. E. MILLER, Assr. to SILMO CHEMICAL CO. (U.S.P. 1,622,390, 29.3.27. Appl., 5.3.26).—Fish oil is added slowly with agitation to an equal weight of fermented milk in an airtight vessel, and the mixture kept for 48 hrs. at 10°. An amount of water equal to that of the oil is added and the whole emulsified. After a further 48 hrs. at 10°, the solid matter is separated from the liquid at 20°, and subsequently powdered. S. S. WOOLF.

Cream-treating process. R. K. COONEY, Assr. to CAMPBELL-COONEY PATENTS CO. (Re-issue 16,576, 22.3.27, of U.S.P. 1,599,650).—See B., 1927, 26.

Butter and cream treating process. R. K. COONEY, Assr. to CAMPBELL-COONEY PATENTS CO. (Re-issue 16,562, 1.3.27, of U.S.P. 1,599,649).—See above.

Treatment of yeasts (E.P. 243,373).—See XVIII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Manufacture of amyl acetate from natural gasoline. G. T. KOCH and G. A. BURRELL (Ind. Eng. Chem., 1927, **19**, 442—445).—A process for the conversion of a 25—40° fraction of gasoline from natural gas, essentially pentane, into amyl acetate is described, the main feature being an ingenious device for the preliminary chlorination of the hydrocarbon in the liquid phase, in the presence of an activated carbon or other porous catalyst. A 90—110° cut of the chlorinated product is then esterified with sodium acetate in an oil-heated autoclave at 205—230° and under a pressure of 200—250 lb., the time of reaction being 8 hrs. The fraction 120—175° of the resulting ester consists of 99—100% amyl acetate ($d^{15.5}$ 0·88) having no moisture, a light yellow colour, and containing 0·196% of free acetic acid. A method of determining the amyl acetate in each fraction is based on the solubility of the ester, and the relative insolubility of the chlorohydrocarbons, saturated ring compounds of the polymethylene series, and paraffin hydrocarbons in sulphuric acid (d 1·84). Cost figures are included derived from the running of a semi-commercial plant.

E. HOLMES.

Distillation of formaldehyde solution. A. ZIMMERLI (Ind. Eng. Chem., 1927, **19**, 524—525).—An apparatus is described in which it was found possible to concentrate solutions containing (*a*) 15·6% of formaldehyde and 3·4% of methyl alcohol, and (*b*) 17·7% of formaldehyde in water, to a point at which the distillate contained 56% of formaldehyde, corresponding to 90% of methylene glycol. This distillate deposits paraformaldehyde immediately on cooling. (Cf. Blair and Taylor, B. 1926, 339.) E. HOLMES.

Humic acids. II. Action of thionyl chloride, of bromine, and of chlorine dioxide on some humic acid samples. W. FUCHS and H. LEOPOLD (Brennstoff-Chem., 1927, **8**, 101—103; cf. B., 1927, 315).—Merck's humic acid and that prepared from quinol behave practically identically when exhaustively treated with thionyl chloride, bromine, and chlorine dioxide, whilst humic acid prepared from starch behaves quite differently. In none of the specimens was any trace of carbohydrates found. Humic acids are feebly reactive substances of high mol. wt., resembling phenols in some respects, but differing from them in their comparatively low content of phenolic hydroxyl groups. W. T. K. BRAUNHOLTZ.

Properties of diethylene glycol. W. H. RINKENBACH (Ind. Eng. Chem., 1927, **19**, 474—476).—A study of the chemical and physical properties of a carefully purified specimen of diethylene glycol has been made, the chief findings being as follows. The bluish crude material loses its colour on purification, and gives an odourless compound with a slightly sweet, burning taste. Densities are given at temperatures between 0° and 35°, that at 15°/15° being 1·1212, whilst the liquid has f.p. $-$ 10·45° $\pm$ 0·05, and b.p. (by extrapolation) 244·5°/760 mm. Calorimetric determinations of the heat of combustion of diethylene glycol gave the values 566·11 kg.-cal./g.-mol. at constant volume, and 566·69 at constant pressure, these values being roughly double those for the corresponding ethylene glycol. The heat of dilution was found to be positive, the compound probably forming a molecular complex with water, having a positive heat of dissolution. It is freely miscible with water, methyl alcohol, ethyl alcohol, ethylene glycol, glacial acetic acid, acetone, furfuraldehyde, pyridine, glycol diacetate, chloroform, nitrobenzene, and aniline, but immiscible with ether, benzene, toluene, carbon disulphide, and carbon tetrachloride, all at ordinary temperatures. Diethylene glycol absorbs more than its own weight of water in 9 days when kept in a closed space over water, but undergoes no hydrolysis. Numerous tables are given, including those for variation of viscosity and refractive index with temperature.

E. HOLMES.

Glycerol tables. L. W. BOSART and A. O. SNODDY (Ind. Eng. Chem., 1927, **19**, 506—510).—New and comprehensive tables are given for the sp. gr. and percentage of glycerol, both apparent and true (*i.e.*, reduced to vacuum), in mixtures of glycerol and water,

based on very careful determinations of sp. gr. of mixtures at 15°/15°, 15·5°/15·5°, 20°/20°, and 25°/25° for every 10%, and for 95 and 97·5% glycerol. The results, calculated to the fifth decimal place, are believed to be more accurate than any hitherto published, agreeing best with those of Nicol. The apparent values now given for 100% glycerol are 1·26557 at 15°/15°, and 1·26362 at 20°/20°, the corresponding true values being 1·26526 and 1·26331. Tables are also given comparing the values given by various workers, and showing the rate of expansion of mixtures between the temperatures 15—20°, 15—25°, and 20—25°. An important correction is noted in a calculation occurring in a paper by Grün and Wirth (A., 1919, ii, 202). E. Holmes.

Moderately dilute sulphuric acid as a reagent for the examination of drugs. H. Teufer (Pharm. Zentr., 1927, **68**, 225—226).—Colour reactions of some vegetable drugs with 70% and 80% sulphuric acid are compared, and the relationship between the colours produced by various barks with acids of the above strengths and the degree of lignification of the cell walls of the barks is discussed. E. H. Sharples.

Occurrence, detection, and determination of ethyl chloride in perfumes. T. Sudendorf and O. Penndorf (Pharm. Zentr., 1927, **68**, 226—228).—A few grams of the ice-cold perfume are added to about 100 c.c. of ice-cold water containing slightly more than the calculated quantity of potassium hydroxide necessary to hydrolyse the ethyl chloride, and the mixture is shaken vigorously at room temperature for at least 5 hrs. About 10 g. of powdered wood charcoal are then added, and, after shaking for 10 min., the liquid is filtered. The filtrate is distilled and the first 5—10 c.c. of distillate are examined for alcohol by the Lieben-Serulla iodoform method or, in doubtful cases, by the Hofmann carbylamine reaction. For the detection of the halogens, an alcoholic solution of slightly more than the calculated amount of potassium hydroxide is added to a few grams of the perfume, both liquids being ice-cold. The mixture is shaken at room temperature, and, if halogens are present, small crystals are deposited on the sides of the vessel. The liquid is decanted, the crystals are dissolved in water, and the solution is examined for halogens by the usual methods. The dangers occasioned by the indiscriminate use of perfumes containing large amounts of ethyl chloride are emphasised. E. H. Sharples.

Genus *Mentha*. XI. Oil of *Mentha Canadensis*, L. H. A. Braun (J. Amer. Pharm. Assoc., 1926, **15**, 337—338).—The greenish-yellow oil (1·16%) had d^{24} 0·8974, α_D + 32·4°; ester 8·06, total alcohols 15·7, free alcohols 7·9, pulegone (R_2SO_3) 20·00, pulegone ($RHSO_3$) 18%. Phenols were absent. Fractionation did not indicate pulegone; the fraction b.p. 140—175° yielded two semicarbazones having m.p. 137° and 180°, respectively.

Chemical Abstracts.

Determination of phenol. Williams.—See II.

Dehydration of sulphite spirit. Schlumberger.—See V.

Medicinal creosote. Bobrov.—See XIII.

***n*-Butyl alcohol and acetone.** Thaysen and Green.—See XVIII.

Patents.

Method of conducting chemical reactions. [Oxidation of acetaldehyde.] C. O. Young and C. J. Herrly, Assrs. to Carbide and Carbon Chemicals Corp. (U.S.P. 1,620,180, 8.3.27. Appl., 20.6.23).—Acetaldehyde is passed in counter-current to a stream of air through a series of vessels at a gradually increasing temperature. The product containing 90% of acetic acid is withdrawn from the last vessel, and the spent air carrying aldehyde vapours is passed from the first vessel through one of two absorbers containing silica gel or other adsorbent at a low temperature. When the adsorbent is saturated it is heated, and the fresh air passing into the apparatus is led through it to recover the aldehyde, the spent air being discharged through the second absorper. T. S. Wheeler.

Manufacture of oxygenated products from hydrocarbons or oxidisable derivatives of hydrocarbons. W. A. Caspari (E.P. 263,201, 11.11.25)—A colloidal solution or suspension of an oxide or oxides of the metals of the fifth or higher group in the hydrocarbon to be treated is atomised with air into a heating chamber at 200—500°. The metallic catalyst is recovered from the condensed product, either unchanged or in the form of the lower oxide. Thus benzaldehyde and benzoic acid may be produced by spraying a colloidal solution of vanadium oxide in toluene with eight times its vapour volume of air, into a chamber at 200—300°. S. Pexton.

Preparation of alkyl hydrogen sulphates. Texas Co., Assees. of R. de M. Taveau (Can. P. 258,227, 12.6.24), —Gases containing olefines are led through a series of absorption vessels containing sulphuric acid in increasing concentration. In the first two vessels, kept at 30°, butylene and propylene, respectively, are absorbed, whilst ethylene is absorbed in the last vessel at 80—120°. C. Hollins.

Production of chlorocarbonates [chloroformates]. J. A. S. Hammond, Assr. to U.S. Industrial Alcohol Co. (U.S.P. 1,618,824, 22.2.27. Appl., 14.7.24).—Gaseous carbonyl chloride (95·5 pts.) is passed into absolute alcohol (58 pts.) at 20°, and the product is treated with water and carbon tetrachloride to separate ethyl chloroformate, which is produced in 90% yield.

T. S. Wheeler.

Preparation of a reactive chloride from pinene or turpentine. F. Höhn (G.P. 426,865, 4.4.25).—Purified turpentine oil or pinene is added in a current of hydrogen chloride, with or without light petroleum, to hydrochloric acid which has been cooled in ice and salt and saturated with hydrogen chloride, giving a very reactive solid chloride, $C_{10}H_{17}Cl$, together with about 30% of an oil from which further quantities of the solid chloride are obtainable by strong cooling. The solid chloride differs from the chloride prepared at temperatures above 15° in that it readily loses hydrogen chloride when warmed, forming camphene. The oil, after separation of the solid, still yields a little camphene when heated. The saturated hydrochloric acid may be used for further batches. C. Hollins.

Preparation of *Bz*-halogenated derivatives of oxindole-3-acetic acid. Chem. Fabr. auf Aktien

(vorm. E. Schering), Assees. of W. Schoeller and K. Schmidt (G.P. 436,518, 11.3.25. Addn. to G.P. 431,510; B., 1926, 870).—Halogenated isatins are condensed with malonic acid, and the unsaturated acids so formed are reduced. Thus, 5-iodoisatin is melted with malonic acid at 130—140° until no more carbon dioxide is evolved, the mass is dissolved in very dilute sodium hydroxide and acidified with dilute acetic acid. The precipitate is filtered off and reduced in sodium hydroxide solution with aluminium amalgam to give 5-iodo-oxindole-3-acetic acid. Dichloro-, dibromo-, and di-iodo-oxindole-3-acetic acids are similarly prepared.

C. Hollins.

Manufacture of acetals. Consortium für Elektrochem. Ind. G.m.b.H. (E.P. 257,622, 27.8.26. Conv., 27.8.25).—The reaction between acetylene and alcohols in the presence of a mercury salt is so conducted that the acetal is removed from the sphere of action as it is formed, *e.g.*, by means of an immiscible solvent, or by distillation, or by " salting out " with potassium acetate, etc. For the preparation of diethylacetal, acetylene is passed rapidly into a suspension of finely-divided mercuric acetate in alcohol containing 10% of sulphuric acid at 75°. Acetal and ethyl alcohol are carried over by the excess of acetylene, and fresh alcohol is added continuously to replace that distilled. C. Hollins.

Preparation of iodinated pyridine derivatives. Deutsche Gold- u. Silber-Scheideanstalt, vorm. Roessler (F.P. 605,226, 20.6.25. Conv., 30.6.24. Cf. E.P. 251,578; B., 1926, 608).—The following are additional. 5-*Iodo-2-hydroxypyridine*, m.p. 191—192°, is precipitated by carbon dioxide from a solution of its sodium salt. 5-*Iodo-2-aminopyridine*, m.p. 129°, from the action of aqueous potassium iodide on aqueous 2-amino-5-diazopyridine, is isolated by making the solution strongly alkaline and distilling in steam. 3-Diazo-2-hydroxypyridine similarly gives 3-*amino-2-hydroxypyridine* (*sodium* salt described).

C. Hollins.

Manufacture of quinoline derivatives. W. Carpmael. From Farbenfabr. vorm. F. Bayer & Co. (E.P. 267,457, 7.9.25).—8-*Nitro-6-methoxyquinoline*, m.p. 159—160°, and 8-*nitro-6-ethoxyquinoline*, obtained by Skraup's reaction from 2-nitro-*p*-anisidine and 2-nitro-*p*-phenetidine, respectively, are reduced by stannous chloride and hydrochloric acid to the corresponding *amines*, m.p. 41° and 60°, and b.p. 137—138°/0·5—1 mm. and 144—145°/1 mm., respectively. The amines are antipyretic, and have also a specific destroying action on blood parasites. C. Hollins.

Manufacture of pharmaceutical products. W. Carpmael. From Farbenfabr. vorm F. Bayer & Co. (E.P. 267,169, 7.9.25).—Strongly basic compounds of the di- and tri-arylmethane, quinoline, phenazine, oxazine, thiazine, acridine, and xanthene series, usually β-dialkylaminoethylamino-derivatives and of pharmaceutical interest, are prepared by a variety of methods from amines of these series. β-Diethylaminoethyl-aniline, b.p. 121—123°/5 mm., is obtained from aniline and β-diethylaminoethyl chloride, and is converted into a thiazine *dye* by oxidation together with *p*-aminodimethylaniline and thiosulphate. The corresponding *methyl-(β-diethylaminoethyl)aniline*, b.p. 124—126°/5 mm., from methylaniline, gives a similar thiazine *dye*. β - *Phenylmethylamino* - δ - *dimethylaminoisopentane*, $NPhMe\cdot CHMe\cdot CHMe\cdot CH_2\cdot NMe_2$, b.p. 133—135°/6 mm., prepared from methylaniline and the appropriate amino-alkyl chloride, gives a thiazine when its *p*-nitroso-compound is reduced and the resulting amine is oxidised with dimethylaniline and thiosulphate. A homologue of malachite-green is obtained by condensing methyl-(β-diethylaminoethyl)aniline with benzaldehyde and oxidising the product. Michler's hydrol is condensed with N-(*β-diethylaminoethyl*)-*acetanilide*, b.p. 134°/3 mm., to give a leuco-base, m.p. 120—125° (decomp.), which is oxidised to a dye base, m.p. 135—138°. *o*-Toluic acid is condensed with methyl-(β-diethylaminoethyl)aniline. From 8-aminoquinoline there are obtained by condensation with β-diethyl-aminoethyl chloride and with β-chloro-δ-dimethyl-amino*iso*pentane, respectively, 8-(*β-diethylaminoethyl*)-*aminoquinoline*, b.p. 180—182°/3—5 mm., and *β-8-quinolinylmethylamino-δ-dimethylaminoisopentane*, b.p. 175 — 183°/7 mm. Similarly 8-amino-6-methoxy-quinoline (cf. E.P. 267,457; preceding abstract) may be condensed with *β-chloro-ε-diethylamino-n-pentane hydrochloride*, m.p. 93° (prepared by reduction of the *methyl γ-dimethylaminopropyl ketone*, b.p. 83—85°, obtained by hydrolysis of *ethyl β-dimethylaminoethyl-acetoacetate*, b.p. 115—120°/5 mm., and treatment of the resulting secondary *alcohol*, b.p. 97°/15 mm., with thionyl chloride), to form β-(6-*methoxy-8-quinolinyl*)*amino-ε-diethylamino-n-pentane*, b.p. 189—190°/2 mm., or with *γ-diethylamino-β-hydroxypropyl chloride hydrochloride* (from epichlorohydrin and diethylamine hydrochloride) to give 8-(*γ-diethylamino-β-hydroxypropylamino*)-6-*methoxyquinoline*, b.p. 225—227°/2 mm. β-(6-*Ethoxy-8-quinolinylamino*)-δ-*dimethylaminoisopentane*, b.p. 204°/mm., is similarly prepared. 4-Chloro-6-methoxy-2-methylquinoline condenses with *N*-methyl-*NN'*-diethylethylenediamine to give 8-(*methyl-β-diethylamino-ethylamino*)-6-*methoxy-2-methylquinoline*, b.p. 179—180°/0·5 mm. δ-Dimethylamino-Δ^{α}-*iso*amylene (G.P. 254,529) is oxidised to the corresponding *oxide*, b.p. 44—46°/17 mm., which condenses with 8-amino-6-methoxyquinoline to form 8-(*δ-dimethylamino-β-hydroxyisoamylamino*)-6-*methoxyquinoline*, b.p. 200—202°/1·5 mm. β-(6-*Methoxy-8-quinolinylamino*)-*ε-dimethyl-amino-n-pentane* has b.p. 196—198°/3 mm. A phenazine *dye* is obtained from *m*-tolylenediamine and the *p*-nitroso-compound of methyl-β-diethylaminoethylaniline. m-(*Methyl-β-diethylaminoethyl*)*aminophenol*, b.p. 156·5—157·5°/1 mm., condenses with *p*-nitrosodimethylaniline to form an oxazine *dye*, isolated as double zinc chloride. From *m*-ethylaminophenol and β-diethyl-aminoethyl chloride is obtained m-(*ethyl-β-diethylamino-ethylamino*)*phenol*, b.p. 180—182°/4 mm.; this condenses with phthalic anhydride to give a *rhodamine*, which may be esterified. m-*Amino-β-diethylaminoethylaniline*, b.p. 158·5—159·5°/1 mm., prepared from *m*-nitroaniline, is converted by the glycerol and oxalic acid method into an acridine *dye*. C. Hollins.

Preparation of derivatives of 6-alkoxy-4-methyl-quinoline. Chem. Fabr. auf Aktien (vorm. E. Schering), Assees. of C. Zöllner (G.P. 436,516, 25.12.24).—α-

Monophthalones of 6-alkoxy-2 : 4-dimethylquinolines are oxidised with chromic acid, permanganate, etc., and the resulting 6-alkoxy-4-methylquinoline-2-carboxylic acids are decarboxylated. 6-*Methoxy*-4-*methylquinoline*-2-*carboxylic acid*, m.p. 220° (decomp.), prepared from 6-methoxy-4-methyl-2-phthalylmethylquinoline, gives 6-*methoxy*-4-*methylquinoline*, b.p. 298°. The corresponding 6-*ethoxy-acid*, m.p. 215—216° (decomp.), and *base*, m.p. 86°, are similarly obtained. C. HOLLINS.

Preparation of vanillin. A.-G. FÜR ANILIN-FABR. (Addn. No. 30,919, 7.9.25, to F.P. 583,856. Conv., 30.10.24. Cf. E.P. 219,676; B., 1925, 26). —4-Hydroxy-3-methoxyphenyltrichloromethylcarbinol, $CCl_3\cdot CH(OH)\cdot C_6H_3(OH)\cdot OMe$, is first oxidised and then hydrolysed, or oxidised and hydrolysed in the same operation. The oxidant must be such as will not attack the aryl nucleus. C. HOLLINS.

Ethyl-*n*-butylbarbituric acid derivatives. E. H. VOLWILER, Assr. to ABBOTT LABORATORIES (U.S.P. 1,621,094, 15.3.27. Appl., 18.7.23).—5-Ethyl-5-*n*-butylbarbituric acid is heated with diethylamine to give *diethylammonium hydrogen* 5-*ethyl*-5-n-*butylbarbiturate*, m.p. about 60°, which is of value as a non-toxic hypnotic. *Di*-n-*butylammonium hydrogen* 5-*ethyl*-5-n-*butylbarbiturate*, m.p. 70°, has similar properties. T. S. WHEELER.

Preparation of arsenical [arsphenamine] solutions. O. LOWY, Assr. to STENECK TRUST Co. (U.S.P. 1,621,121, 15.3.27. Appl., 20.12.20. Cf. U.S.P. 1,559,899; B., 1926, 108).—Diaminodihydroxyarsenobenzene dihydrochloride is treated in solution with 4·3—8 g.-mols. of sodium hydroxide, and kept out of contact with air for at least 4 hrs. to permit of complete formation of the disodium salt of the arsenobenzene derivative. *E.g.*, it is sealed in an ampoule in an atmosphere of nitrogen until required for use. Solutions so prepared are non-toxic. T. S. WHEELER.

Concentration of dilute acetic acid. H. SUIDA (U.S.P. 1,624,810—1, 12.4.27. Appl., 18.6.24. Conv., 26.6.23).—See E.P. 218,271—2; B., 1925, 899.

Manufacture of ester condensation products. R. M. CARTER, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (Re-issue 16,591, 12.4.27, of U.S.P. 1,472,324, 30.10.23).—See B., 1924, 77.

Manufacture of menthol. K. SCHÖLLKOPF, Assr. to RHEINISCHE KAMPFER-FABR. G.M.B.H. (U.S.P. 1,625,771, 19.4.27. Appl., 28.11.22. Conv., 23.11.21).—See E.P. 189,450; B., 1923, 743 A.

Manufacture of halogen substituted oxindole-3-acetic acids and homologues thereof. W. CARPMAEL. From CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING) (E.P. 266,539, 27.2.26).—See G.P. 433,099; B., 1927, 286.

Manufacture of *OO*-diacyl derivatives of diphenolisatin. E. PREISWERK, Assr. to HOFFMANN-LA ROCHE CHEMICAL WORKS (U.S.P. 1,624,675, 12.4.27. Appl., 18.10.23. Conv., 27.11.22).—See E.P. 221,976; B., 1925, 26.

Phenols from ammoniacal liquor (G.P. 436,522).—See III.

Butyric acid etc. (U.S.P. 1,625,732).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic sensitivity. T. S. PRICE (J.S.C.I., 1927, **46**, 145—150 T).—A review of recent work.

Effects of dilution and stirring of a photographic developer. A. H. NIETZ and R. A. WHITAKER (J. Franklin Inst., 1927, **203**, 509—535).—See B., 1927, 60.

Flame movement. ELLIS.—See II.

PATENTS.

Photographic film and base. S. E. SHEPPARD and S. S. SWEET, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,623,500, 5.4.27. Appl., 27.4.26).—The film consists of a sensitive layer, such as a gelatino-silver halide emulsion, a flexible colloidised supporting layer of a cellulose compound, and between them and firmly attached to both, anon- strippable elastic waterproof cushioning stratum, such as a composition containing rubber, preferably depolymerised, and mixed with a resin. W. CLARK.

Production of coloured photographs on paper or the like. E. A. LAGE (U.S.P. 1,623,123, 5.4.27. Appl., 25.7.21. Conv., 30.7.20).—See E.P. 188,692; B., 1923, 73 A.

Films non-sensitive to electrical action (F.P. 611,136—7).—See V.

XXII.—EXPLOSIVES; MATCHES.

Solubility of nitroglycerol in water. W. LEDBURY and C. W. FROST (J.S.C.I., 1926, **46**, 120 T).—Nitroglycerol, present in aqueous solution, can be determined by a method which involves the reduction of the nitrogen of the ester to ammonia. Data for the solubility of nitroglycerol in water have been obtained over a temperature range of 15° to 80°, and show that the solubility is not a linear function of the temperature. The solubility at 15° is approximately 0·13 g. per 100 c.c., whilst that at 80° is 0·35 g. per 100 c.c. The increase in solubility with temperature is most marked at the higher temperatures. No appreciable hydrolysis of the ester occurred over the temperature range in question.

PATENTS.

Triazo-nitrate [azidonitrate] explosives. Explosive composition. F. H. BERGEIM, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,620,714—5, 15.3.27. Appl., [A], 18.6.26; [B], 9.9.26).—(A, B) β-Azidoethyl alcohol is nitrated with a mixed acid containing 60% of sulphuric and 40% of nitric acid, to give β-*azidoethyl nitrate*, which is more sensitive and more powerful than nitroglycerin, and can be employed as a substitute for it. T. S. WHEELER.

Primer for cartridges. O. C. HORNEY (U.S.P. 1,619,867, 8.3.27. Appl., 17.7.25).—The primer consists of Monel metal. T. S. WHEELER.

Determination of the effect of explosives. E. SCHMILAUER (Austr. P. 104,411, 6.3.25).—The intensity of the action of explosives is determined by the effect of firing a charge in a lead cylinder made in two symmetrical parts, surrounded by a tube. L. A. COLES.

Brisant explosive. B. J. FLÜRSCHEIM (U.S.P. 1,624,401, 12.4.27. Appl., 9.10.24. Conv., 26.10.23).—See E.P. 226,913; B., 1925, 268.

XXIII.—SANITATION; WATER PURIFICATION.

Chlorination reduces foaming in Imhoff tanks. C. COHEN (Eng. News-Rec., 1927, **98**, 563—564).—At Lufkin, Texas, the addition of lime to the vents and sludge digestion chambers reduced "foaming" only to a slight extent. With a view to inhibition of the organisms which give rise to foaming and elimination of bad odours, chlorination of the raw sewage was tried. A reduction of the rate of foaming was soon noticeable, and became more so with continued application of chlorine at the rate of 6 p.p.m. After a few weeks prechlorination was discontinued, and lime treatment recommenced, but foaming recurred. Feeding the chlorine directly into the sludge-digestion chambers was then tried without success. On reverting to prechlorination of the sewage, satisfactory conditions were again established. Latterly, foaming has been controlled by the application of chlorine during the period 8.0 a.m. to 5.0 p.m. only, at the rate of 3 p.p.m. of chlorine calculated on the daily flow. The tank effluent contains no residual "free" chlorine, and is of better quality than formerly. W. T. LOCKETT.

Super-chlorination of [water to remove] chlorophenol tastes. L. B. HARRISON (J. Amer. Water Works Assoc., 1927, **17**, 336—340).—Chlorine was added in increasing amounts to water containing 0·075 p.p.m. of phenol, and the solutions were kept for 30 min. Chlorophenol tastes made their appearance with 0·125 p.p.m. of chlorine, were not noticeable with 0·5—0·75 p.p.m., but again appeared with 0·8—0·95 p.p.m. With 1·0—2·0 p.p.m. no chlorophenol tastes were detected. 0·8 p.p.m. of chlorine was sufficient to destroy the chlorophenol tastes produced by wood distillation wastes, and 1·2 p.p.m. those produced by gas wastes, using concentrations of wastes of 1 : 10,000. Experiments, in which taste was observed after dechlorination indicated that the minimum time required for the reaction between chlorine (1·2 p.p.m.) and phenolic waste is at least 2 hrs. With a low temperature a larger dose of chlorine is required and a longer period of reaction. (Cf. Howard and Thompson; B., 1927, 204.) W. T. LOCKETT.

Prevention of phenol taste with ammonia. J. W. MCAMIS (J. Amer. Water Works Assoc., 1927, **17**, 341—350).—In the water supply of Greeneville, Tenn., phenol-like tastes developed during the summer months, some considerable time after chlorination. The origin of the tastes could not be definitely established. The conditions obtaining precluded largely the possibility of the presence of industrial wastes and the contamination of the water with impure air. Algal growth was not responsible. Elimination of the taste has been successfully brought about at very low cost by treatment of the water with ammonia, along the lines of the Metropolitan Water Board's experiments, prior to chlorination. W. T. LOCKETT.

Moderns methods of sewage disposal. W. BUTLER and J. H. COSTE (J.S.C.I., 1927, **46**, 49—59 T).—The efficacy of sedimentation processes is limited by the slow rate of fall of fine particles and by the fact that much of the matter, which should be removed from sewage or altered chemically before a satisfactory effluent can be discharged, is in solution. Land treatment can yield satisfactory results under some conditions, but easily drained land is costly in this country. The intensive biological treatment of sewage can effect in a few hours a degree of purification which takes much longer on land, and it is in biological processes that the solution of the sewage problem is to be found. The activated sludge process has been studied, and a comparison made between the results obtained by air diffusion or blowing methods as used by Fowler and agitation processes which have been applied with success by Haworth, Bolton, and others. The authors consider that air diffusion is particularly suitable for re-aerating used activated sludge—an essential part of the cycle—whilst mechanical agitation is better adapted for actual treatment. In both types of plant it was found that the quantity of sewage effectively treated was increased three- or four-fold if the greater part of the suspended matter was previously removed by sedimentation. Analyses of activated sludge are given, and experimental work is described which shows that, notwithstanding the great part played by air in the changes produced, an immediate purification, yielding in some cases a stable effluent, occurs when activated sludge and sewage or sedimented sewage are mixed. Experiments are also adduced showing the advantages of re-aeration of activated sludge and the small effect of variations in the depth of diffuser tanks.

Dissolved oxygen absorption test [of sewage]. E. A. COOPER and W. H. READ (J.S.C.I., 1927, **46**, 154—156 T).—In experiments with the object of accelerating Winkler's dissolved oxygen absorption test, evidence of an increased oxygen absorption on the addition of potassium dihydrogen phosphate to the sewage has been obtained. Other alkali phosphates have little or no effect. Potassium dihydrogen phosphate causes an increase in the number of bacteria in the sewage, but the addition of cultures of sewage organisms has an inappreciable influence on dissolved oxygen absorption. Gelatin adsorbs a greater amount of phosphate from solutions of the foregoing potassium phosphate than from equimolecular solutions of other phosphates. The specific effect of the potassium salt on dissolved oxygen absorption is thus not merely due to its capacity of increasing the number of bacteria present, but is more likely to be due to an increased metabolism of the individual bacteria, owing to the more ready availability of the phosphate.

Chemical oxidation of the constituents of sewage. Action of hydrogen peroxide. E. A. COOPER and W. H. READ (J.S.C.I., 1927, **46**, 156—157 T).—Hydrogen peroxide oxidises certain constituents of sewage, but there is no advantage in employing a hydrogen peroxide test instead of the Forschammer permanganate process

in the analysis of sewage effluents. The catalytic action of ferrous sulphate on the thermal decomposition of hydrogen peroxide is retarded by the presence of sewage, and this has been found to be due to the ammonia and primary amines present. The presence of one part per 100,000 of hydrogen peroxide diminishes the dissolved oxygen absorption of the sewage effluents, owing apparently to an inhibitory effect on the bacteria.

Destruction of germs by ammonia and chlorine. W. OLSZEWSKI (Chem.-Ztg., 1927, **51**, 269—270).—The results of the bacteriological and chemical examination of water in a swimming bath, after treatment with chlorine and ammonia, are described. The gases, which are used in the ratio of 2 pts. of chlorine to 1 pt. of ammonia by wt., probably owe their bactericidal effect to chloroamine. Compared with the use of chlorine alone, the method has the advantage that no odour of chlorine is perceptible, the water remains clearer, and a careful regulation of the amount to be added is not required. F. R. ENNOS.

Chemical action of *p*-quinones on proteins. E. A. COOPER and S. D. NICHOLAS (J.S.C.I., 1927, **46**, 59—60 T).—It is confirmed that whilst *p*-benzoquinone is a very much more active germicide than toluquinone, yet there is little difference in their reactivity towards gelatin and other proteins. Charcoal and asbestos adsorb the two quinones equally, so that the foregoing results may be due to the disturbing effect of the adsorption. With cystine, alanine, and creatinine, the difference in the reactivity of the two quinones is also surprisingly small, suggesting that the anomalous results with proteins are not entirely caused by adsorption, but have also in part a structural explanation. On the other hand, benzoquinone reacts 4—5 times as rapidly with glycine and asparagine as toluquinone, thus giving results similar to those obtained in the bacteriological work. The observations suggest that simple water-soluble substances may be of as much fundamental importance in the life of the cell as the more complex colloids, upon which greater emphasis is usually placed.

Water analysis. D. BURTON and J. K. HASLAM J.S.C.I., 1927, **46**, 111—114 T).—The methods for determining temporary, permanent, and magnesia hardness and oxygen in boiler feed water are critically examined so that sufficiently accurate results for control purposes can be obtained in softening water supplies to the usual limit of about 3° hardness. The suitability of various indicators for determining the temporary hardness is discussed. Pfeifer and Wartha's method is shown to give reliable results for the permanent hardness providing a Munktell No. 1 F filter paper is used and the water contains no alkali carbonate. High results are obtained for the magnesia hardness due to (1) carbonation of the lime-water, (2) removal of lime by the acid required to change the colour of the indicator in removing the temporary hardness, (3) precipitation of lime from the lime-water because its solubility decreases with rise in temperature.

Significance of nitrogen determinations in sanitary analysis. S. L. NEAVE and A. M. BUSWELL (J. Amer. Water Works Assoc., 1927, **17**, 388—395).—A discussion as to the value of the free and albuminoid ammonia and total organic nitrogen figures obtained in the routine analysis of domestic sewage. Analyses have been made both by routine methods and by determinations of the nitrogenous components of the samples using the methods of the physiological chemist. In the latter the actual ammonia was aspirated into standard acid, the carbamide then converted into ammonia by adding urease, and the resulting ammonia similarly aspirated into standard acid, the difference between total (Kjeldahl) nitrogen and the sum of the nitrogen contained in the ammonia and carbamide being taken as the protein fraction. The analyses indicated that "free ammonia" as obtained by distillation represents actual ammonia, a small amount of amine, and a part (5—20%) of the carbamide; and "albuminoid ammonia" represents an indefinite portion (25—55%) of the carbamide and a very small amount of the protein fraction. A revision of the accepted methods for determining the nitrogen of sewage, with a view either to correcting their defects or abandoning them in favour of more significant determinations, is recommended. W. T. LOCKETT.

Bactericidal action of selenium and tellurium compounds. STOVER and HOPKINS.—See XVI.

PATENTS.

Production of base-exchange substances. COCHRANE CORP., Assees. of S. DAHL-RODE (E.P. 245,092, 11.12.25. Conv., 23.12.24).—Ground furnace slag containing silicates of aluminium and alkaline-earth metal is treated with a hot strong solution of caustic alkali, heated until substantially all the water has evaporated, dried, and graded. Granules of the required size are then leached to remove excess alkali, and subsequently treated with a dilute mineral acid or dilute acetic acid to remove the remaining alkali and undesirable calcium compounds. Finally the product, preferably after washing, is treated with brine, washed, drained, and air-dried. W. T. LOCKETT.

Apparatus for regenerating zeolites and the like. W. H. GREEN, Assr. to GENERAL ZEOLITE Co. (U.S.P. 1,620,886, 15.3.27. Appl., 17.7.22).—A tank of regenerating solution has a pipe communicating with the treating vessel below the pervious bed of treating material, and a return pipe from the treating vessel to drain the space above the reaction bed. Suitable discharge pipes and valves for the waste are fitted to the reaction vessel. W. G. CAREY.

Product for destroying mosquito larvæ. E. C. C. ROUBAUD and R. A. VEILLON (U.S.P. 1,625,627, 19.4.27. Appl., 21.11.23. Conv., 30.11.22).—See E.P. 207,802; B., 1924, 804.

Filters [for sewage etc.] (E.P. 266,857).—See I.

Hydrocyanic acid for fumigation (U.S.P. 1,620,365).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 3, 1927.

I.—GENERAL; PLANT; MACHINERY.

Apparent specific gravity and porosity. Comparison of various methods. G. M. Nave (J.S.C.I., 1927, **46**, 158—159 T).—Seven methods of finding the apparent specific gravity and porosity of porous material such as firebrick or coke are briefly described and criticised. Preference is given to a method the principle of which is to coat the material with just melted paraffin wax. The specific gravity bottle method, with xylene as the liquid, is recommended for the determination of the real specific gravity using material which passes at least a 1/100 in. mesh sieve.

Patents.

Impact pulveriser. A. B. Willoughby (U.S.P. 1,622,695, 29.3.27. Appl., 31.1.25).—The material is entrained in two opposing co-axial jets of superheated high-pressure steam and pulverised by impact of the material against itself. Coarse material is allowed to drop out of the zone of impact on to a bottom wall which is inclined towards an outlet, which is not directly below the jets. Fine material is removed from the pulveriser casing by an ejector. B. M. Venables

Pulveriser. H. E. Witz, Assr. to Babcock & Wilcox Co. (U.S.P. 1,621,571, 22.3.27. Appl., 25.5.23. Conv., 26.5.22).—The material passes through a pulveriser proper, an expansion chamber, and an exhaust fan. Oversize is separated out in both the separating chamber and the fan casing, and is returned to be re-ground. B. M. Venables.

Granulating dry material. F. L. Bryant (U.S.P. 1,621,869, 22.3.27. Appl., 12.7.24).—A number of disintegrator discs are mounted on a common vertical shaft and increase in diameter downwards. The material, after suffering impact in one zone, drops to the next larger zone, All the particles are substantially in suspension, and not subjected to attrition with consequent heating. B. M. Venables.

Apparatus for evaporating solutions. J. L. ter Hall (G.P. 436,240, 17.5.22. Conv., 2.3.22. Addn. to G.P. 393,408; B., 1924, 698).—In a tubular evaporator, the compartment below the tubes is divided into two sections by a horizontal partition to which are attached narrow tubes discharging into the lower ends of the main tubes. L. A. Coles.

Vacuum evaporator. L. P. Devaucelle (F.P. 612,548, 2.7.25).—In lead-lined iron evaporators, both sides of the lining are under vacuum. L. A. Coles.

Air-cleaning apparatus. F. F. Pease, Assr. to F. F. Pease, Inc. (U.S.P. 1,620,780, 15.3.27. Appl., 5.4.23. Renewed 29.7.26).—The air to be cleaned is admitted at one end of a chamber through a port surrounding a rotary atomiser for liquid. Liquid and dirt are collected on a rotary separating screen at the other end of the chamber, and the clean air is removed by a fan. B. M. Venables.

Apparatus for separating liquids and gases. Tray. K. W. Bartlett (U.S.P. 1,620,750—1, 15.3.27. Appl., 6.5.26).—Forms of bubbling trays for rectifiers are described. B. M. Venables.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Mechanism of coking. E. Audibert and L. Delmas (Fuel, 1927, **6**, 131—140, 182—189; Chim. et Ind., 1927, **17**, 355—366; cf. B., 1926, 569).—The mechanism of the swelling ("intumescence") of coal has been studied by heating a cylindrical pellet, prepared by compressing coal dust, and observing the changes in length which occur. The temperature at which intumescence occurs is always higher than the temperatures, at the same rate of heating, of initial softening, and of initial evolution of volatile matter by thermal decomposition. The slower the rate of heating the less is the amount of swelling; if the rate is so slow that fusion is incomplete no swelling is observed. All the coals studied were capable of intumescence if the rate of heating was sufficiently rapid. With very rapid heating intumescence begins about 25° lower than with rates of heating in the neighbourhood of 1° per min. Intumescence is caused by bubbles of the gaseous decomposition products, which are imprisoned in the plastic mass, and is therefore only an apparent expansion. It can be prevented by the addition of an infusible powder to the coal, the amount required depending on the nature of the coal, the fineness of the coal and of the added material, the apparent density of the mixture, and the rate of heating. There is no direct relationship between the rate of evolution of volatile matter and swelling; a modification of the rate of heating which causes the suppression of swelling may be accompanied indifferently by an increase or a decrease in the rate of evolution of volatile matter. The swelling of a coal is suppressed by preheating *in vacuo* at 350° for $4\frac{1}{2}$ hrs., or by oxidation in air at 120° for 4 hrs. The structure of the coke produced depends on the rate of heating of the coal over the range 350—500°, the crushing strength passing through a maximum value as the rate of heating is increased. To produce coke of satisfactory strength the filling up of the interspaces between the particles of coal, which takes place in each layer of the charge as it passes through the plastic state, should be sufficiently

nearly complete without exceeding the limit giving maximum compactness. If the obstruction of the interspaces becomes too great bubbles are formed, and the resulting coke has a weak and porous structure. The optimum conditions can be attained, not only within the ordinary range of coking coals, but by suitably blending two coals neither of which is itself capable of yielding a good coke. A. B. Manning.

Laboratory study of the formation and structure of coke. D. J. W. Kreulen (Fuel, 1927, **6**, 171—182).—The transformation of coal into coke (*a*) during a volatile matter determination, (*b*) by the sudden heating of a piece of coal, and (*c*) in the Fischer–Schrader test is described and illustrated by sections of the material at different stages of the process. Varying the rate of heating in the Fischer–Schrader apparatus produces variations in the amount of swelling; beginning with very rapid rates, the swelling shows first an increase as the rate falls, reaches a maximum, and then decreases. The coke made from the largest sized particles of coal is the most porous and fragile. Attempts to transform a semi-coke into a high-temperature coke by subsequently heating to 1100° gave a solid coke with, however, a large number of fissures. With addition of graphite to the coal the crushing strength of the coke passes through a maximum value. The increase of strength is greater the smaller the particle size of the added material, but the maximum increase in all cases is obtained by addition of approximately the same surface area of inert matter per unit of coal. Addition of sand shows the same phenomenon, except that two maxima are obtained. Microscopical examination of the coke indicates that the structures observed are similar to the cenosphere structures described by Newall and Sinnatt (B., 1925, 87; 1926, 905). A. B. Manning.

Industrial transformation of bituminous coal into technical products. G. Patart (Fuel, 1927, **6**, 160—170; cf. B., 1927, 66).

Influence of an anti-knock compound in a gas-ion oxidation. S. C. Lind and D. C. Bardwell (Ind. Eng. Chem., 1927, **19**, 231—233).—A study was made of the influence of selenium diethyl on the slow oxidation of methane at ordinary temperatures under the ionising influence of radon. A mixture of methane with 2 vols. of oxygen was saturated with selenium diethyl, and samples were diluted to give mixtures 0·001, 0·01, and 0·046 molar with respect to selenium diethyl. These were enclosed in glass reaction spheres of 2 cm. diameter with radon, and the oxidation was measured manometrically and checked chemically. The kinetics were calculated by the general equation $dP/dt = k\mu E_t P$, the rate of pressure change being proportional to two variables, the pressure, P, and amount of emanation, E_t, present at any time, t, and to the constant of ionisation, k, and μ, an efficiency factor for converting ionisation into chemical action. The molecules reacting per pair of ions in the mixture should be six, which was not attained in the mixture without anti-knock, but was slightly exceeded in the 0·01 molar mixture. Thus the presence of anti-knock did not retard the reaction, and ionisation probably plays no important part in explosive reaction or flame propagation. Anti-knock increases the velocity of flame propagation. It is suggested that non-selective absorption dependent on the absolute density of the gas mixture may affect flame propagation. H. Moore.

Comparison of gasolines by analytical and engine tests. D. R. Stevens and S. P. Marley (Ind. Eng. Chem., 1927, **19**, 228—231).—The anti-knocking properties of 18 samples of gasoline of widely different types were investigated. These were analysed by Egloff and Morrell's method (cf. B., 1926, 570), and the values for naphthenes, unsaturated hydrocarbons, and aromatics reduced to an aromatic equivalent. The gasolines were then tested on a modified Delco lighting 1250-watt engine with adjustable spark timing. In an indicator of the bouncing-pin type the shock of detonation was indicated by a long lever on a scale, a standard fuel being made to give a reading in the middle of the scale, a compression ratio of 5·5 : 1 being employed. To test the accuracy of the analysis synthetic mixtures were made up, and in the engine trials the standard fuel was blended with enough benzene or kerosene to bring its degree of detonation to correspond with the fuel under trial. Ricardo rates the amounts of olefines, naphthenes, and aromatics to give the same effect in reducing detonation as 5 : 4 : 1, but a proportion of 2 : 2 : 1 is more nearly correct in the case of methyl*cyclo*hexane, hexylene, and toluene. There was wide divergence in many cases between the results of analysis and engine trials. Anti-knock values can only reliably be determined by engine trials. H. Moore.

Oxidation of petrol by air. M. Freund (Z. angew. Chem., 1927, **40**, 368—374).—A mixture of petrol vapour with air or nitrogen was passed through a heated iron tube charged with petroleum coke and the exit gases were analysed. It was found that the degree of decomposition increased with the temperature up to 500° and with the proportion of air in the mixture. With four successive passages of a given quantity of petrol vapour through the tube at 400° about 30% was converted into oxidation products, including carboxylic acids, phenols, aromatic and unsaturated and cyclic aliphatic hydrocarbons. Tests with nitrogen in place of air indicated that pyrogenous decomposition also occurs. Water was also produced to the extent of 4—5%, and was found to contain acetic acid in solution. The permanent gases resulting from the process were found to contain 0·8% of carbon dioxide and no oxygen. C. Irwin.

Utilisation of acid tar from the purification of illuminating oil and other oils. M. E. Schmitz (Sealed note 2038, 28.9.10. Bull. Soc. Ind. Mulhouse, 1927, **92**, 693—695).—Acid tars are mixed at 80° with a maximum of 30% of crude xylol, b.p. 130—200°, and after remaining for at least 5 hrs. sulphuric acid, *d* about 1·530, is withdrawn, and can be used for treating fresh quantities of oil. The residual tar, after washing with water or dilute alkali, is distilled to recover the solvent and asphalt. Acid tar obtained from illuminating oil is preferably mixed with 2 pts. of the tar from heavier oils before treatment. L. A. Coles.

Utilisation of acid tars. M. E. Schmitz (Sealed note 2049, 11.11.10. Bull. Soc. Ind. Mulhouse, 1927, **92**, 695

—698).—Acid tars from which sulphuric acid has been recovered by the process described in the preceding abstract are distilled to coke if not suitable for the production of asphalt. " Black " sulphuric acid obtained by concentrating to 92% strength the recovered acid, by pouring it slowly into boiling acid *d* 1·84, is more efficacious for purifying mineral oils than is pure acid.
L. A. Coles.

Production of asphalt from acid sludge from the purification of mineral oils. M. E. Schmitz (Sealed note 2173, 9.5.12. Bull. Soc. Ind. Mulhouse, 1927, **92**, 698—702).—Terpenic hydrocarbons of b.p. up to 250°, obtained by distilling with superheated steam the acid resin precipitated by diluting with water the acid tar from the purification of illuminating oil, are suitable for diminishing any loss of solvent incurred in the processes already described (see preceding abstracts). The acid resin need not be previously distilled, but may be added to the acid tar together with the solvent, or, alternatively, its precipitation may be dispensed with by mixing 1 pt. of acid tar from illuminating oil with 2 pts. of tar from heavier oils before addition of solvent. L. A. Coles.

Report on the processes described by M. E. Schmitz in sealed notes nos. 2038, 2049, and 2173. G. Hugel (Bull. Soc. Ind. Mulhouse, 1927, **92**, 702—706. See preceding abstracts).—Recovery of acid is less complete than that claimed, 2—5% remaining in the tar. The solubility of acid resins in crude xylol may be very low, in which case higher-boiling fractions of b.p. 200—300° can be used as solvent. The cost of the tar-oil fractions is probably too great to render the process of commercial value. L. A. Coles.

Oil shales of Tasmania. A. McI. Reid (Proc. Roy. Soc. Tasmania, 1926, 42—51).—Tasmanite, which occurs in the North and North-Central districts of Tasmania associated with kerogenite and humic kerogenite coals, was examined. Oil is not originally present in tasmanite, being generated by heat from the contained kerogen. The shale stands weathering well, and ignites and burns readily. Its yield of oil is 40—45 gals./ton. Analysis showed 30·84% of volatile matter and 62·50% of ash. The crude oil contained 79·34% C, 10·41% H, 4·93% O, 0·31% N, and 4·93% S, and had *d* 0·931—0·956. The crude oil yielded on distillation 10% of benzine and 80% of fuel oil, and from the latter 25% of lighting oils and 30% of lubricating oils were obtainable, these being of asphaltic base. The benzine is rich in aromatics, and a good anti-knock motor fuel. H. Moore.

Patents.

Method of breaking coal. P. G. Seigle and G. E. Dean (U.S.P. 1,620,838, 15.3.27. Appl., 2.1.25).—The production of fines is avoided by breaking the coal under water. T. S. Wheeler.

Purifying gases from sulphur. F. Fischer and H. Tropsch (E.P. 254,288, 3.6.26. Conv., 29.6.25).—Gas containing organic sulphur compounds and hydrogen is passed over a catalyst consisting of finely-divided lead or tin or alloys thereof, deposited upon an inorganic substance (*e.g.*, bases or acid anhydrides) which remains solid at the temperature of reaction, *i.e.*, above the m.p. of the metallic catalyst. Such a contact material, consisting of equal parts of lead chromate and copper oxide treated with hydrogen at 400°, is able at 300—500° and ordinary pressure to desulphurise gas and maintain its activity for several months. S. Pexton.

Destruction of carbon monoxide in the waste gases from internal combustion engines. C. W. White (F.P. 612,282, 6.2.26).—The gases are oxidised by air in the presence of a catalyst. A. B. Manning.

Gas producers. T. M. Davidson, and Patent Retorts, Ltd. (E.P. 263,932, 5.10.25).—A machine comprising a gas producer and carbonising plant is described. The producer consists of two vertical retorts superimposed upon a water-gas generator. Between the two retorts are provided two supplementary vertical retorts which terminate above the generator and have laterally disposed discharge openings. Surrounding all the retorts are combustion flues, in which the " blow gases " from the water-gas generator are burnt, for providing the necessary heat for carbonisation. Coal fed into the producers is completely gasified, whilst the coal fed into the supplementary retorts yields high-grade gas and coke. S. Pexton.

Method of producing gas. O. U. Bean, Assr. to Bean Process Syndicate, Inc. (U.S.P. 1,621,652, 22.3.27. Appl., 24.3.25).—Superheated steam, dissociated into its chemical elements, is mixed with hydrocarbon vapour, and the mixture subjected to alternate expansion and contraction and increase of heat to a temperature at which the hydrocarbons combine with the hydrogen to produce a mixture of gases.
C. O. Harvey.

Recovery of condensable vapours from gases. H. H. Armstrong (U.S.P. 1,621,821, 22.3.27. Appl., 11.7.23).—A liquid absorbent is atomised by, and passes through an enclosed space in contact with, the gas. Thence the mixture passes into a chamber where it comes into intimate contact with unatomised liquid absorbent which is circulated through the chamber.
C. O. Harvey.

[Low-temperature] carbonisation by direct internal heating. Deutsche Erdöl-A.-G. (G.P. 435,517, 24.12.22).—Bituminous material is carbonised by internal heating with a regulated hot mixture of combustion gases and cold combustible gases, so arranged that the carbonisation takes place in parallel with an independent main current of combustible gases, from which the amount required for the carbonisation is withdrawn, and to which after carbonisation the gases are returned, the return being made at a point either before or after the point of withdrawal. A. B. Manning.

Retort. G. McD. Johns (U.S.P. 1,621,785, 22.3.27. Appl., 12.11.23).—Finely-divided flowable material (*e.g.*, coal) flows down a vertical narrow space between two parallel walls, one of which is heated and is not perforated; the other is composed of a number of transversely corrugated plates, set obliquely, and overlapping so that spaces are left for the exhaust of distillate, the material being maintained in a thin layer against the heated wall. B. M. Venables.

Conversion [cracking] of oils. J. C. Clancy (U.S.P. 1,620,075, 8.3.27. Appl., 16.4.24).—A molten

mixture of zinc chloride (80%) and sodium chloride (20%) is passed at 400—600° slowly down a vertical retort filled with nickel packing units. High-boiling oil containing from 5—10% of aluminium chloride in suspension is introduced into the retort through a washer, in which it is met by the mixed vapours of oil and aluminium chloride coming from the retort. The chloride vapours and the higher-boiling oils are here condensed and conveyed back to the retort. Any carbon formed in the cracking process is removed, together with uncracked oil, by the descending stream of molten chloride. To reduce the amount of carbon separating, hydrogen is passed into the retort. The apparatus can also be employed to treat the sludges resulting from the distillation of oils with aluminium chloride. T. S. Wheeler.

Treatment of hydrocarbon oils. R. C. Holmes, Assr. to Texas Co. (U.S.P. 1,623,729, 5.4.27. Appl., 17.8.21).—The oil is cracked by passage once only through a tube maintained at 370—480°, and under a pressure of 500 lb. or more, and being thus maintained in continuous liquid phase, coke production is substantially avoided. Separation of the lower- from the higher-boiling fractions is effected in an expansion chamber at substantially reduced pressure connected with a reflux condenser. C. O. Harvey.

Fuel oil. F. L. Dietz (U.S.P. 1,620,635, 15.3.27. Appl., 3.1.22).—A fuel for internal-combustion engines comprises low-temperature tar (35 pts.), tetralin (30 pts.), and alcohol (35 pts.). T. S. Wheeler.

Apparatus for treating crude oils. H. A. Hopkins (U.S.P. 1,621,782, 22.3.27. Appl., 16.2.25).—Crude hydrocarbon oils are cleansed and dehydrated in an apparatus consisting of upper and lower oil chambers containing heating pipes and connected by means of a series of open-ended tubes, the oil passing from the lower to the upper chamber and outwardly therefrom. C. O. Harvey.

Treatment [cleaning] of hydrocarbon oils. L. E. Jackson (U.S.P. 1,619,869, 8.3.27. Appl., 4.10.23).—Fouled naphtha from dry-cleaning operations is treated under agitation with a quantity of potassium hydroxide solution (*d* 1·23) exceeding by 10% that required to neutralise the aliphatic acids present, together with activated charcoal and a small quantity of ethyl alcohol. Rapid separation of the impurities present takes place. T. S. Wheeler.

[Sludge-removal] treatment of oils. E. A. Evans, and C. C. Wakefield & Co., Ltd. (E.P. 267,174, 5.10.25).—The sludging tendencies of transformer oils are reduced by the addition of 0·1—2% of a neutral soap of aluminium, calcium, copper, lead, nickel, tin, or zinc, or of a metallic salt of a fatty acid (*e.g.*, sodium acetate). The soap may or may not be soluble in the oil. C. O. Harvey.

Refining mineral oils with liquid sulphur dioxide. A.-G. für Chem. Ind. m.b.H. (E.P. 267,071, 14.5.26. Conv., 5.3.26).—Economies in heat and size of condenser are effected in an apparatus for recovery by evaporation of the liquid sulphur dioxide, by passing the extract through one or more evaporators operating at pressures higher than the condenser pressure, of which evaporators only that working at the highest pressure is heated externally, the others deriving their heat from the condensation of the sulphur dioxide vaporised in the previous evaporator. The extract is finally heated in a still at condenser pressure. A portion of the vapour from the first evaporator may be branched off and used for heating elsewhere. C. O. Harvey.

Apparatus for condensing oils. C. B. Buerger, Assr. to Gulf Refining Co. (U.S.P. 1,622,941, 29.3.27. Appl., 18.7.24).—The vapours are delivered into a tower containing a column of water in circulation at a point above the column, and the condensate is withdrawn from the top of the column, means being provided for equalising the pressure below and above the point of introduction of the vapours. C. O. Harvey.

Method of dephlegmation. J. D. Seguy, Assr. to Universal Oil Products Co. (U.S.P. 1,623,136, 5.4.27. Appl., 25.3.25).—The capacity of a dephlegmating column is varied at will by withdrawing the reflux condensate and introducing the vapours at predetermined vertically-spaced points, the vapours being introduced at a point above the liquid level in the column. C. O. Harvey.

Oil still. J. E. Bell, Assr. to Sinclair Refining Co. (U.S.P. 1,623,773, 5.4.27. Appl., 10.8.23).—In an apparatus for heating an oil-cracking still a portion of the hot waste heating gases which have previously passed over the heating surfaces of the still is mixed with the products of combustion before again passing them over the heating surfaces, and air, preheated by heat interchange with waste heating gases, is supplied to the firebox. C. O. Harvey.

Still. H. M. Lasher, Assr. to Kansas City Gasoline Co. (U.S.P. 1,622,452, 29.3.27. Appl., 1.7.20).—A still for cracking petroleum hydrocarbons is fitted with an internal rack the suspensions for which pass through stuffing boxes in the top of the still, and are thus operable from without. C. O. Harvey.

Ionising retort (E.P. 267,358).—See XI.

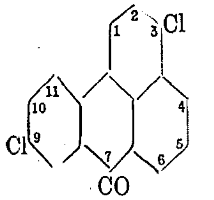
IV.—DYESTUFFS AND INTERMEDIATES.

Patents.

Preparation of vat dyes. I. G. Farbenind. A.-G., Assees. of A. Lüttringhaus, H. Wolff, and H. J. Emmer (G.P. 436,887, 12.10.24).—By the action of hypochlorous acid on benzanthrone there are obtained two chlorohydroxybenzanthrones, m.p. 356° and 262—263° respectively. These are precipitated by acids from alkaline solution, and are separated by crystallisation from acetic acid. They are not identical with *Bz*-chloroderivatives of 1-, 2-, or 3-hydroxybenzanthrone. They are methylated by means of methyl toluenesulphonate, and by subsequent fusion with alkali give navy-blue vat dyes. C. Hollins.

Preparation of dyes of anthracene series. I. G. Farbenind. A.-G., Assees. of A. Lüttringhaus and H. Wolff (G.P. 436,533, 1.3.25).—The halogenated compounds of G.P. 193,959 are condensed in the presence

of sodio-arylamines (sodio-aniline) to give violet vat dyes. Calcium, magnesium, and other metals may be substituted for sodium. Chlorobenzanthrone is heated with a solution of sodio-aniline in aniline. 3 : 9-*Dichlorobenzanthrone* (annexed formula), m.p. 263—264°, prepared from 9-chlorobenzanthrone and sulphuryl chloride, gives 9 : 9′-*dichloro*iso*dibenzanthrone.* 11 : 3-*Dichlorobenzanthrone*, m.p. 218—220°, obtained from 11-chlorobenzanthrone, m.p. 174° (from 1-chloroanthraquinone), and sulphuryl chloride, similarly gives 11 : 11′-*dichloro*iso*dibenzanthrone.* C. HOLLINS.



Preparation of vat dyes of the anthraquinone series. I. G. FARBENIND. A.-G., Assees. of F. KAČER (G.P. 436,535, 25.1.25).—Anthraquinonethiazoles are nitrated, and, if desired, the nitro-compounds are reduced and acylated. The nitro- and amino-anthraquinonethiazoles are grey-brown to red-brown vat dyes, the acylaminoanthraquinonethiazoles intense yellow to orange-yellow. C. HOLLINS.

Manufacture of writing inks. A. G. BLOXAM. From I. G. FARBENIND. A.-G. (E.P. 267,633, 23.12.25).—Writing inks fast to water are made by adding alkali (50 c.c. of $2N$-sodium hydroxide per litre) or formaldehyde, or both, to a solution of a suitable azo dye. The writing becomes fast to water after keeping for a period (1 hr. to several days), which varies according to the dye used and the sizing on the paper. The presence of free amino- or hydroxyl groups in the dye is advantageous. Suitable types of dye are :—(*a*) [*m*-Diamine ← *p*-diamine]$_2$ → aminonaphtholsulphonic acid. (*b*) Diaminodiphenylamine → [H-acid → *m*-diamine]$_2$. (*c*) *p*-Diamine → amine → aminonaphtholsulphonic acid. C. HOLLINS.

Preparation of pigment dyes. J. P. H. ØRS (Dan. P. 34,077, 25.9.23).—Basic dyes are precipitated by means of aqueous sodium silicate on the calcium silicate, alumina, barium sulphate, calcium carbonate, kaolin, lithopone white, titanium white, or other appropriate substrate. C. HOLLINS.

Manufacture of dyes. R. GANSSEN and G. GÖRZ (G.P. 435,804, 15.3.24).—Humus tars which are otherwise unsuitable for colouring purposes are converted into chestnut-brown products by treatment with chlorine gas or mixtures of liquors capable of liberating chlorine. A. J. HALL.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Viscose. X. G. KITA, R. TOMIHISA, K. SAKURADA, and H. KONO (J. Cellulose Inst., Tokyo, 1927, **3**, 27—31).—The influence of the temperature in the preparation and maturing of the alkali-cellulose was studied. The viscose from alkali-cellulose treated at 25° is easily spread on a glass plate, but the film becomes detached in the setting bath, whereas the viscose from alkali-cellulose prepared at 0° is not easily spread. The tenacity and especially the extension of the film are adversely affected by storage of the alkali-cellulose at 25°, whereas they are not affected by storage at 0° and in both cases they increase similarly to a certain extent during the ripening of the viscose. With alkali-cellulose treated at 8—10° the viscose ripened at 0° is not easily spread, and requires some time in the setting bath, giving a white translucent film, whereas viscose ripened at 20° is readily coagulated giving a semi-transparent film. Ripening at 20° induces spontaneous coagulation of the viscose after 5 days. When the viscose is ripened at 0° the tenacity and extension do not as a rule increase with the time of ripening. Alkali-cellulose aged for 5 days gives the highest results. Ripening the viscose at 20° gives films equal in tenacity and extension to those from viscose ripened at 0°. Alkali-cellulose prepared with 13% caustic soda lye gives inferior films to that prepared with 25% or 30% lye ; the viscose does not spread easily, the films are less transparent, and the tenacity and extension are poor. J. F. BRIGGS.

Viscosity of cellulose solutions. C. R. GENUNG (Ind. Eng. Chem., 1927, **19**, 476).—A modification in Small's cuprammonium method (B., 1925, 495) for the determination of the viscosity of cellulose solutions is proposed, involving the use of a more positive system for flushing out the apparatus with hydrogen and filling with reagent, and the limitation of the range of copper concentration of the cuprammonium solution to 2·95—3·05%. The method is claimed as more accurate and easier of manipulation than the original. E. HOLMES.

PATENTS.

Degreasing of wool and other textile materials. N. V. ALGEM. CHEM. PRODUCTENHANDEL (G.P. 435,753, 30.3.24. Conv., 26.1.24).—Wool is degreased by treatment with organic solvents, the latter being removed while the wool is continuously led through a closed chamber maintained under diminished pressure so that air or other gas enters the chamber where the wool enters and leaves it. The air saturated with vapour of the solvents is cooled below 0° and led through adsorption chambers for recovery of the solvents. A. J. HALL.

Degreasing wool. N. V. ALGEM. CHEM. PRODUCTENHANDEL (Dutch P. 15,202, 20.9.24).—Thin layers of wool carried on a travelling brattice within a closed chamber are sprayed with an organic solvent. A. J. HALL.

Protecting animal fibres during treatment with alkaline solutions. I. G. FARBENIND. A.-G., Assees. of K. DAIMLER, G. BALLE, and F. JUST (G.P. 434,979, 21.12.23).—Sulphonic or carboxylic acids of resinous products obtained by condensing aromatic hydrocarbons with, *e.g.*, halogenated hydrocarbons, alcohols, or sulphur chloride, are added to alkaline solutions for treating animal fibres. L. A. COLES.

Production of fibres, silk, hair, films, etc. [from viscose]. KÖLN-ROTTWEIL A.-G. (F.P. 612,879, 17.3.26).—The products are prepared from fresh viscose containing unmatured alkali-cellulose, and to the viscose and/or the precipitation bath is added material having the property of decreasing the rapidity of the action of the spinning bath, such as condensation products prepared from formaldehyde and naphtholsulphonates or naphthalenesulphonates, or sulphonated condensation products from vegetable resins and phenol, or material

such as sulphite-cellulose waste liquor, carbazolesulphonates, sulphonated coumarone-resin, or aromatic sulphonic acids. L. A. COLES.

Production of cellulose ethers. E. TEUPEL (G.P. 435,346, 29.4.22. Addn. to G.P. 408,342; B., 1925, 397).—The earlier process is rendered more rapid and economical by using 25—40% alkali hydroxide solution containing salt. L. A. COLES.

Cellulose composition [containing ββ'-dichloroethyl ether]. W. G. LINDSAY, Assr. to CELLULOID CO. (U.S.P. 1,620,977, 15.3.27. Appl., 2.6.24).—ββ'-Dichloroethyl ether is of value as a solvent for cellulose ethers or esters, and can be used for the manufacture of varnishes, lacquers, and the like. T. S. WHEELER.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Manufacture of copper sulphate. II. The system copper sulphate–sulphuric acid–water. G. AGDE and H. BARKHOLT (Z. angew. Chem., 1927, 40, 374—379; cf. B., 1926, 707).—The solubility curves for copper sulphate and sulphuric acid at temperatures from 0·5—80° were determined, and also curves of solutions of varying composition but of similar density. Addition of sulphuric acid always reduces the solubility of copper sulphate. Beyond a limit which varies with the temperature the pentahydrate is converted into the trihydrate. Cooling curves on the solubility diagram show the increase in sulphuric acid content of a saturated acid–copper sulphate solution during crystallisation. In large-scale operation, when fresh acid is added to the mother-liquor for the treatment of cupreous material in a tower, the regulation of temperature and concentration so that the limits of transition from pentahydrate to trihydrate are not overstepped has effected a 40% increase in output from a given plant. The crystals may be freed from acid by washing with saturated copper sulphate solution on the centrifuge. Contrary to general opinion, however, crystals from acid solution do not necessarily effloresce in air, though they may be distinguished by their more fissured appearance. C. IRWIN.

Commercial preparation of aluminium acetate. F. CHEMNITIUS (Chem.-Ztg., 1927, 51, 210).—Aluminium sulphate with a maximum content of 0·02% Fe, 70—80% acetic acid free from organic impurities, and finely-divided calcium carbonate are the raw materials used. 360 kg. of aluminium sulphate are dissolved in 300 litres of water with direct steam heating. 450 litres of water are added and 146 kg. of acetic acid calculated as 100%. Then 192 kg. of calcium carbonate are added in small portions with vigorous stirring. After 240 hrs. 680 litres of water are added and the mixture is allowed to remain for 6 days, with at first occasional agitation. The decanted and filtered liquor (*d* not less than 1·048) is freed from heavy metals and iron by the successive addition of barium sulphide and potassium ferrocyanide, the excess of the latter being removed with zinc sulphate, followed by a further addition of barium sulphide. The settled solution is filtered through paper and animal charcoal. Later development of turbidity in the clear colloidal solution is due to the presence of precipitating salts, especially of iron, which must be carefully removed, as above stated. C. IRWIN.

PATENTS.

Recovery of carbonate compounds of sodium and borax from brines. W. A. KUHNERT (U.S.P. 1,618,835, 22.2.27. Appl., 20.8.25. Cf. U.S.P. 1,618,834; B., 1927, 252).—Brine is concentrated and treated with carbon dioxide at 30—45° until all but 5% of the sodium carbonate present has been precipitated as the compound, $Na_2CO_3,NaHCO_3$. It is then filtered and cooled at 20°, when 50—75% of the borax present separates. T. S. WHEELER.

Preparation of iron carbonyl. I. G. FARBENIND. A.-G., Assees. of C. MÜLLER and K. HUBERICH (G.P. 436,369, 13.2.25).—Iron carbonyl is prepared by the action of carbon monoxide on iron at ordinary or higher pressures, the product, which is deposited on the iron, being distilled off from time to time under reduced pressure. A. B. MANNING.

Manufacture of iron carbonyl. M. MÜLLER-CUNRADI, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,614,625, 18.1.27. Appl., 27.5.26. Conv., 5.1.25).—Carbon monoxide is caused to act upon iron under pressure and at a temperature sufficiently high to promote the formation of iron carbonyl without decomposition of the carbon monoxide. H. ROYAL-DAWSON.

Production of iron carbonyl. I. G. FARBENIND. A.-G. (F.P. 608,415, 28.12.25. Conv., 9.2.25).—Carbon monoxide and iron are brought together, if necessary under raised pressure, in a reaction chamber connected directly with a condensing chamber maintained at a lower temperature. L. A. COLES.

Manufacture of aluminium chloride. E. R. WOLCOTT, Assr. to TEXAS CO. (U.S.P. 1,617,696, 15.2.27. Appl., 22.9.21).—A lining for retorts used in the manufacture of aluminium chloride consists of a mixture of clay and an alkali silicate.

Manufacture of aluminium chloride. G. L. PRICHARD and H. HENDERSON, Assrs. to GULF REFINING CO. (U.S.P. 1,616,549, 8.2.27. Appl., 3.3.22).—Hydrated bauxite is mixed with heavy hydrocarbon material, *e.g.*, asphalt, at a dehydrating temperature, and the mixture is carbonised and exposed to the action of chlorine. H. ROYAL-DAWSON.

Production of sodium aluminate. J. B. BARNITT, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,616,674, 8.2.27. Appl., 21.1.26).—An intimate porous mixture of an aluminous material, soda ash, and fuel is formed, and, while the component parts are in a quiescent condition, the fuel is burned to produce a sintered mass of sodium aluminate. H. ROYAL-DAWSON.

Manufacture of chromates from chromium ores. I. G. FARBENIND. A.-G. (F.P. 608,928, 4.1.26).—Mixtures of chromium ores with calcium hydroxide and sodium carbonate are treated in thin layers with oxidising combustion gases. L. A. COLES.

Production of manganese arsenate. GRASSELLI CHEMICAL CO., Assees. of W. L. TANNER (Can. P. 260,934, 29.7.24).—Intimate mixtures of manganese compounds with arsenious oxide are heated in the presence of an oxidising agent, or, alternatively, arsenious oxide is

treated with manganese compounds having an oxidising action, in the presence of water. L. A. Coles.

Production of a hard body from asbestos. Siemens & Halske A.-G. (E.P. 241,576, 16.10.25. Conv., 17.10.24).—Asbestos is heated just below its fusing point, and while in this condition is subjected to high pressure between rollers, thus yielding a mass capable of being machined. H. Royal-Dawson.

Making aqueous solutions and the product. A. M. Shook, Assr. to Aquazone Corp. (U.S.P. 1,616,202, 1.2.27. Appl., 16.10.23. Conv., 9.6.23).—The solvent is freed from gas-forming nuclei, and solutes are introduced into it, *e.g.*, disodium hydrogen phosphate, sodium bicarbonate, sodium sulphate, sodium chloride, and potassium bicarbonate, any gas-forming nucleus being avoided. Oxygen is then introduced, under conditions of temperature and pressure favourable to absorption, to produce supersaturated solutions. H. Royal-Dawson.

Calcining magnesite. Magnesit-Industrie A.-G. (Austr. P. 104,404, 7.3.24).—Magnesite is heated in furnaces, preferably of the rotating tube type, in such a manner that the temperature near the source of heat is higher than that required to calcine the magnesite to the desired extent, and, if necessary, is sufficiently high to dead-burn it. The product is withdrawn from the zone in which it has been calcined sufficiently to give it the desired properties. L. A. Coles.

Recovery of vanadium. K. B. Thews, Assr. to Colorado Vanadium Corp. (U.S.P. 1,621,038, 15.3.27. Appl., 29.10.20. Renewed 9.8.26. Cf. U.S.P. 1,430,864; B., 1922, 901 a).—Crude non-alkaline solutions of soluble vanadates obtained from vanadium ores are treated with sodium chlorate, or other oxidising agent, and with calcium chloride solution. A precipitate of pure calcium vanadate separates. T. S. Wheeler.

Manufacture of lead oxide. J. J. Tardan (F.P. 611,810, 30.1.26).—Lead sponge, prepared by the action of zinc on acidified solutions of lead salts or by electrolysis of alkaline solutions containing lead salts, is stirred for 24—48 hrs. in wooden or cement vessels whereby it is oxidised by the air practically completely to the monoxide. A. R. Powell.

Manufacture of oxide of lead. H. Waring, and Associated Lead Manufrs. Ltd. (E.P. 267,191, 12.11.25).—After passing air through molten lead by the usual method, any incompletely oxidised lead is violently agitated and comminuted in a heated oxidising atmosphere by a high-speed impact pulveriser of special construction having a peripheral speed of at least 20,000 ft./min. W. G. Carey.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Specific heat of iron. P. Oberhoffer and W. Grosse (Stahl u. Eisen, 1927, **47**, 576—582).—From measurements of the specific heat of electrolytic iron between 0° and 1590° the transformation points have been found to be: A2 ($\alpha-\beta$), 785°; A3 ($\beta-\gamma$), 906°; and A4 ($\gamma-\delta$), 1401°; and the m.p. 1528°. The absorption of heat on heating the metal is 6·765 g.-cal./g. at A3, 2·531 g.-cal./g. at A4, and 64·38 g.-cal./g. at the m.p. The curve showing total heat–temperature is convex to the temperature axis up to the A2 point, and this fact, coupled with the absence of any heat effect at this temperature, shows that the A2 transformation is proceeding regularly throughout the range 0—785°, and is not of an allotropic nature. The total heat–temperature curves for the β- and δ-ranges are portions of the same straight line, showing that the atomic configuration of both forms is the same. The curve for γ has a smaller slope towards the temperature axis than that for β and δ. The mean specific heat of pure iron rises from 0·1107 between 0° and 100° to 0·1628 between 0° and 780°, at which figure it remains constant to 900°, when there is a sharp rise to a maximum of 0·1724 at 0—906°, followed by a slow fall to 0·1632 at 0—1400°. The mean specific heat between 0° and the m.p. is 0·2071. Calorimetric researches with a transformer iron containing 4% Si show that this alloy does not undergo the A3 transformation on heating. The true specific heat rises rapidly from 0·118 at 100° to 0·248 at 750° (A2 point) then falls abruptly to 0·185 between 755° and 1476°. Between 1476° and 1488°, which is the melting range of this alloy, a heat absorption of 62·209 g.-cal./g. takes place; the specific heat of the molten alloy is 0·141. A. R. Powell.

Determination in advance of the composition of the flue gases and the blast requirements of cupolas [in the cast-iron foundry]. B. Osann (Stahl u. Eisen, 1927, **47**, 533—537).—It is shown from theoretical considerations and confirmed by experiment that in a properly run cupola melting iron for casting, the correct volume of air required for the blast and the composition of the flue gases are functions of the proportion of coke in the furnace charge, other constituents of the charge being constant. The oxygen content of the flue gases decreases linearly with the coke content of the charge from 1·5% with 11% of coke, to zero with 14% of coke. Using a charge with 6% of limestone and assuming in all cases that 4·7 kg. of coke per 100 kg. of charge are burnt to carbon dioxide, and the remainder to carbon monoxide, then the proportion of the first-named gas in the flue gases decreases linearly from 13·5% with 11% of coke, to 10% with 16% of coke, while the proportion of carbon monoxide increases from 12 to 17% and the hydrogen content remains more or less constant at about 3%. The volume of air required in cubic metres is $k[8\cdot8+0\cdot93(b-4\cdot7)]$, where b is the carbon content of the charge, and $k=5\cdot0$ for less than 14% of coke and 4·76 for more than this. A. R. Powell.

Bright dipping of metal [copper and brass]. A. K. Graham (Trans. Amer. Electrochem. Soc., 1927, **51**, 219—228).—The application of acid dips to copper and brass, and the acid mixtures frequently recommended are discussed. A study of the chemistry of dips containing sulphuric, hydrochloric, and nitric acids shows that the rate of dissolution of copper varies directly with the nitric acid concentration, whilst the rate of dissolution of zinc varies directly with the concentration of hydrochloric acid. The activity of a dip depends upon the concentration of sulphuric acid and upon the temperature. The rate of dissolution of copper increases up to 50°, but that for zinc decreases; above this tempera-

ture all dips decompose rapidly. Results obtained on copper and zinc separately have been found to apply to both high and low brass. Operating details for bulk dipping of metal are given. Chemical control of dips and mechanical dipping are discussed.

M. E. Nottage.

Manufacture of magnesium. D. B. Keyes (Trans. Amer. Electrochem. Soc., 1927, **51**, 159—166).—Attempts have been made to produce magnesium by dissolving magnesium oxide in some suitable electrolyte and then electrolysing. Preliminary experiments carried out with a large number of salts of the alkali and alkaline-earth metals, aluminium, and zinc showed that positive results could be obtained only with those electrolytes which contained either barium or calcium fluoride as one of the constituents. In the process finally adopted, the bath consisted chiefly of magnesium fluoride, together with small quantities of calcium, barium, and sodium fluorides. A specially-designed cell was used in which the electrodes were set at an angle of 45° to the horizontal in a graphite-lined container; by this means the horizontal movement of the electrolysis current was obtained without passing the electrodes through the sides of the furnace. Under suitable electrical conditions there was no indication of the decomposition of either magnesium or calcium fluoride, provided that an excess of magnesium oxide was present. The magnesium obtained was free from undesirable impurities.

M. E. Nottage.

Pure zinc. H. M. Cyr (Trans. Amer. Electrochem. Soc., 1927, **51**, 169—173).—"Spectroscopically pure" zinc was prepared by a series of fractional distillations *in vacuo* of chemically pure zinc at relatively low temperatures. The product contains less than 0·001% of metallic impurities and shows no impurity lines when examined spectrographically. The metal shows unusual resistance to tarnishing and retains its bright lustre even after exposure to ordinary air for two years. It also shows a great resistance to the action of hydrochloric and sulphuric acids; the purity of the acids is, however, an important factor. The slow rate of dissolution in these acids can be hastened by the use of a platinum couple. Nitric acid attacks "spectroscopically pure" zinc much faster than the commercial grades. Comparison of the resistance to acids of the original crystals with the worked or recast metal shows that most of the resistance is due to its purity rather than to its original shape. The metal, m.p. 419·45°, is unusually soft, has a very low recrystallisation temperature, a tensile strength of 1000 kg./cm.^{2}, and possesses the same ductility and elongation as commercial zinc but less temper elasticity. It undergoes no allotropic change between its m.p. and normal atmospheric temperature.

M. E. Nottage.

Preparation of compact and colloidal metallic molybdenum. E. Wedekind and O. Jochem (Z. angew. Chem., 1927, **40**, 434—438).—Metallic molybdenum in lumps weighing up to about 8 g. may be obtained by heating the dioxide or trioxide with calcium in a vacuum. Reduction with zinc yields the metal in the form of a powder, which can be peptised by the method previously described for the peptisation of zirconium (A., 1908, ii, 501). The particles of the resulting hydrosol, which keeps well if air is excluded, are negatively charged, are immediately coagulated by alkaline electrolytes and more slowly by neutral electrolytes, but are comparatively resistant to hydrogen ions.

R. Cuthill.

Patents.

Steel alloy. J. W. Weitzenkorn, Assr. to Molybdenum Corp. of America (U.S.P. 1,622,894, 29.3.27. Appl., 30.3.26).—A steel alloy contains up to 2·50% C, 0·50—1·45% Cr, 0·10—0·50% V, up to 0·48% Mo, and traces of impurities.

M. E. Nottage.

[Iron–chromium] alloy. E. J. Shackelford, W. B. D. Penniman, and J. R. Cain, Assrs. to Radiac Metals, Ltd. (U.S.P. 1,622,977, 29.3.27. Appl., 16.4.25).—The process consists of adding ferrochromium to a furnace charged with material producing basic slag and molten iron having up to 0·05% C, working the bath until the carbon content is under 0·3%, adding a second charge of ferro-chromium, and repeating this step until the bath has the desired chromium and carbon content.

M. E. Nottage.

Chromium plating. Metals Protection Corp., Assees. of H. C. Pierce (E.P. 267,080, 23.8.26. Conv., 5.3.26).—Articles of iron, steel, or nickel are cleaned electrolytically in an alkaline bath, and after washing are made the anode in an electrolyte prior to plating in a chromium bath. During plating it is sometimes found beneficial to alternate the polarity of the metal to promote a more even deposition of chromium on the surface of articles in relief.

C. A. King.

Silver alloys. W. M. Guertler (E.P. 267,263, 21.1.26).—An alloy of silver, copper, and/or manganese, aluminium, magnesium, and tin, together with one of the auxiliary hardening metals, zinc, cadmium, tin, antimony, and magnesium.

C. A. King.

[Iron] mould for casting metals. W. H. Smith, Assr. to Ford Motor Co. (U.S.P. 1,621,472, 15.3.27. Appl., 14.8.25).—A mould for hot metal casting is made of iron containing less than 1% of other elements.

T. S. Wheeler.

Metallising insulating sheets or bands. Dubilier Condenser Co., Ltd., Assees. of E. Pfiffner (E.P. 263,728, 26.2.26. Conv., 2.1.26. Addn. to E.P. 243,393).—The material is coated alternately with a metallic salt solution and a reducing agent by spraying the reducing agent, mixed with compressed air or steam, on to the band wetted with the salt solution, and drying or washing the band after spraying before its reintroduction into the salt solution.

W. G. Carey.

Treatment of minerals, slags, or the like. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 267,433, 10.11.26).—The metals contained in minerals, which are attacked only with difficulty, may be successively eliminated in a grinding plant either by modifying the temperature of working, or the concentration of the liquid decomposing agent, or by employing different decomposing agents. Any gases evolved during the decomposition may be continuously withdrawn from the reaction chamber.

M. E. Nottage.

[Discharge valve for solids from] mineral concentrating plant. A. France (U.S.P. 1,616,791, 8.2.27. Appl., 26.9.23. Conv., 14.3.23).—In apparatus

such as upward-current classifiers, the material of highest sp. gr. is discharged by a device comprising a flap valve at the top diverting the material to either of two chambers, the bottom of each of which comprises an outlet flap valve. Each compartment near the top is provided with an inlet for liquid and a vent pipe.

B. M. Venables.

[Ore crushing] mill. W. W. Gibson (U.S.P. 1,621,775, 22.3.27. Appl., 25.8.24).—A rotary shaft carries crushing cylinders evenly spaced around the shaft and equidistant from it, and crushing members are provided in each cylinder. A scoop is secured to the shaft for picking up ore during the rotation, and tubes of equal length each connect the scoop to one of the cylinders.

H. Holmes.

XI.—ELECTROTECHNICS.

Patents.

Methods and apparatus for the electrical purification of gases. Siemens-Schuckertwerke G.m.b.H. (F.P. 609,259 ; 609,422 ; 609,426 ; 609,728 ; 609,765—6 ; 609,880—1 ; 609,932—6 ; 609,985—7 ; 609,989 ; 610,139—610,142 ; 610,181—5, 13.1.26—26.1.26. Conv., 14.1.25—18.12.25).—(a) Gases containing sticky materials in suspension are treated, prior to their entrance into the purifier, with oils, tetralin, water, steam, etc. in such proportions that the suspended matter is carried down with the precipitating vapour in the form of a liquid. (b) Electrode insulators have the form of hollow cylinders, and are fixed without the use of screws or bolts. (c) The propagation of an explosive wave in an electrical gas purifier is prevented by a device whereby the explosion pressure operates a mechanism which damps out turbulence of the dust in the purifying chamber. (d) Electrodes are spaced so close together that the dust cloud does not overstep the explosive limit. (e) Tubes, gliding planes, etc. are arranged in the gas-way in order to deliver the dust from the electrodes to the collecting chamber. (f) Electrode insulators are fixed at one end and carry the electrodes at the other end, so that the points of support are below the bearing surface of the electrodes. (g) The temperature of the gas to be treated is automatically controlled in the plant by a device inserted in the gas stream. (h) The point of entrance of a high-tension electrode is made conical, a sheet of asbestos etc. being tightly inserted between the electrode and the wall of the opening so that rupture of the insulation may be prevented. (i) In plant designed more especially for the separation of dust from gases derived from the low-temperature distillation of coal, the dust-separating chamber is heated so that no condensation of tar vapours occurs therein. (j) High resistances are inserted in the high-tension conductors in order to produce a uniform distribution of current. (k) The gas stream traverses the purifying chamber, passing across a part of the space between the electrodes, first from above downwards, and then from below upwards. (l) The gas stream flows between electrodes arranged in a shaft, passing normally between the upper opening of the shaft and the roof thereof. (m) A dynamo-electric machine produces directly the direct high-tension current employed. (n) Gases are cooled to the appropriate temperature by a cooling medium, *e.g.*, water, in tubes. (o) In order to effect uniform distribution of the gas stream between the electrodes, a device is provided to produce turbulence before or in the lower part of this interval. (p) The precipitating surfaces are constructed and disposed in accordance with the pressures in the gas stream. (q) One or more circuit breakers, each comprising a coil, are placed in the high-tension conductor. (r) In an electrical precipitating plant designed for the treatment of hot gases, especially moist gases, the walls are made double, so that hot air may be passed between the walls. (s) Electric waves, producing interrupted sparks in the treatment chamber, control apparatus indicating the passage of sparks, or reducing the electrode potential. (t) Gas is passed through the purifying chamber before and, if necessary, after the precipitating process, in order to render the precipitation more regular. (u) An object, *e.g.*, an iron plate, is placed above the gas entrance to the treatment chamber, in order to distribute the gas uniformly during its ascent through the chamber. (v) Prior to entering the purifier, gases are brought into intimate contact with metals resting on the floor of the purifier for the removal of ions. (w) Discharge electrodes are arranged on the floor of the purifier, whilst the pendant, insulated, precipitating electrodes are connected with the source of high-tension current. (x) Under each precipitating electrode a "guttiform object" is arranged, which collects dust falling from the respective electrodes and delivers it to a collector. (y) A precipitating electrode is made in the form of a wooden frame, having active electrode material on both sides separated by a partition, and openings for removal of dust. (z) A precipitating electrode is made of a hollow wooden frame, the two active parallel surfaces being provided with openings and the edges being arranged so that they can be introduced, either partly or wholly, into the gas stream. Means are provided for transferring precipitated dust into the interior of the frame electrode.

J. S. G. Thomas.

Insulating compartment in electrical gas purifiers. Metallbank u. Metallurgische Ges. A.-G. (G.P. 434,829, 8.5.25). — The insulators are kept clean by a current of air drawn through the insulator compartment by the heat from the inner compartment of the purifier.

L. A. Coles.

Electric furnace. J. C. Woodson, Assr. to Westinghouse Electric and Manuf. Co. (U.S.P. 1,622,621—2, 29.3.27. Appl., 23.6.25).—A hanger block forming the sole support for a resistor member extends into and away from the face of a member of substantially overhung channel section, and is held in proper operative position therein by the resistor.

J. S. G. Thomas.

[Heating resistances for] electric furnaces. Internat. General Electric Co., Inc., Assees. of Allgem. Elektricitäts Ges. (E.P. 262,796, 9.12.26. Conv., 9.12.25).—Free, self-supporting spirals of chromium–nickel alloy, having an internal diameter not greater than about 8 times that of the wire of which they are made, are hung both horizontally and vertically in the heating chamber.

J. S. G. Thomas.

Electric accumulator. H. Leitner (E.P. 267,205,

7.12.25).—Hard cement-porous blocks made of a mixture of a paste of lead oxide and liquids causing the paste to set, *e.g.*, tar products, glucose, etc., are cast in moulds around metallic conductors. Powdered pumice or substances which can subsequently be removed, *e.g.*, sulphates of magnesium, potassium, or sodium, etc., are incorporated in the block in order to make it sufficiently porous to absorb electrolyte within its pores without impairing the conductivity of the mass.

J. S. G. THOMAS.

[Active material for] electric accumulators. H. LEITNER (E.P. 267,588, 14.12.25).—Finely-divided metallic particles of lead are sprayed on to a conductor and treated electrolytically to form a closely adherent, porous layer of lead peroxide. The process is also applicable to the preparation of plates for other accumulators, *e.g.*, nickel-iron-alkali cells. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 24,353 of 1893 and 4431 of 1882.] J. S. G. THOMAS.

Electrolytic cell. A. A. JAHNKE (U.S.P. 1,623,246, 5.4.27. Appl., 20.8.25).—Anode and cathode electrodes, and a pair of gas-collecting chambers placed one within the other and permitting accumulation of gases and gradual increase of gas pressure, are arranged within a housing. J. S. G. THOMAS.

Ionising retort [for gasifying hydrocarbons etc]. I. W. HENRY (E.P. 267,358, 28.5.26).—A retort is supplied with means for creating and maintaining in the retort chamber during operation a non-discharging, non-sparking, high-frequency alternating field operating solely by induction, and effecting physicochemical changes in the material treated. The retort is provided with a valve-controlled inlet and a gas conduit. J. S. G. THOMAS.

[Electrolytic] method and means for coating materials [*e.g.*, metal wire] with rubber. KODAK, LTD., Assees. of S. E. SHEPPARD and L. W. EBERLIN (E.P. 261,700, 17.5.26. Conv., 20.11.25).—See U.S.P. 1,589,329; B., 1926, 794.

Filter diaphragms for electrolytic purposes. J. BILLITER, and SIEMENS & HALSKE A.-G. (E.P. 245,127 and 262,470, 21.12.25. Conv., 23.12.24).—See F.P. 608,309; B., 1927, 196.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Pitch, turpentine oil, and aqueous distillates during the industrial treatment. P. A. BOBROV (Trans. Viatka Sci. Res. Inst., 1926, 2, 52—67).—The method of distillation of turpentine followed in one of the numerous Viatki factories is described, and figures are given to show how the turpentine oil etc. vary during the process of distillation. T. H. POPE.

Medicinal creosote and methods of obtaining it. P. A. BOBROV (Trans. Viatka Sci. Res. Inst., 1926, 2, 87—133).—The literature is summarised, and it is shown that when red turpentine is distilled in a current of steam, the distillate furnishes a material suitable for the preparation of medicinal creosote or guaiacol. The distillate is purified by treatment with sodium hydroxide solution, and filtration of the alkaline solution through an absorbent such as charcoal removes the hydrocarbons and other extraneous substances dissolved along with the phenols. T. H. POPE.

Characteristics of the process of rosin distillation. P. A. BOBROV (Trans. Viatka Sci. Res. Inst., 1926, 2, 68—81).—This process is regarded as consisting of a number of simultaneous or successive changes, the essential one being the liberation of the galipot, the volatile compounds of which distil as oil of turpentine, whilst the non-volatile components, notably the resin acids, remain in the residual pitch. At the same time the turpentine undergoes pyrogenetic changes, the pinene being converted partially into dipentene, and aliphatic and aromatic hydrocarbons formed. At higher temperatures the lævorotatory resin acids are transformed into dextrorotatory isomerides, and these acids are partly converted into esters of phenols and partly decomposed by heat giving mainly hydrocarbons.

T. H. POPE.

Composition of resin. P. A. BOBROV (Trans. Viatka Sci. Res. Inst., 1926, 2, 40—51).—Owing to the inconsistency of the results obtained when pine resin is distilled either by direct heating or in a current of steam, the method of extracting with ether and investigating the ethereal solution has been employed (cf. Tschirch, A., 1903, i, 105, 106). In this way the constituents of the resin have been separated into the following groups: free acids; a mixture composed principally of phenols; 37·8—38·1% of substances of indifferent character, such as hydrocarbons, aldehydes, ketones, resins, etc., 1·7% consisting of phenolic esters; and saponifiable substances. The fixed acids, amounting to 43·4—47·5% of the resin, consisted of 21·7% of aliphatic acids and 73·67% of resin acids, 6·8% being hydroxy-acids. Crystallisation from light petroleum and alcohol yields an acid $C_{20}H_{30}O_2$, m.p. 161—161·5°, $[\alpha]_D + 32°$ (in alcohol). The proportion of non-volatile phenols in the resin was 6—8·17%, and 0·927—0·99% of the resin was insoluble in ether. T. H. POPE.

Pine resin acids and their behaviour at high temperatures. P. A. BOBROV (Trans. Viatka Sci. Res. Inst., 1926, 2, 29—39).—A summary of the literature.

T. H. POPE.

PATENTS.

Preparation of ethereal non-resinous condensation products from phenol and formaldehyde. A RIEBECK'SCHE MONTANWERKE A.-G. (G.P. 436,445, 15.1.21. Addn. to G.P. 352,003; B., 1922, 728 A).—The initial condensation product of a phenol with formaldehyde in the presence of an acid catalyst is further treated with vinyl or allyl halides in alkaline solution. The vinyl or allyl ethers of dihydroxydiarylmethane thus formed are clear, viscous, transparent liquids solidifying on exposure to the air. S. S. WOOLF.

Production of higher aldehyde derivatives of reaction products of aldehydes and amines. C. O. NORTH (U.S.P. 1,619,953, 8.3.27. Appl., 5.3.26).—The product obtained by the condensation of acetaldehyde (3 mols.) with aniline (3 mols.) is dehydrated at 105°, heated under reflux for 14 hrs. with about 60% of its weight of acetaldehyde, and then at 105°. A hard resinous product is obtained. T. S. WHEELER.

Writing inks (E.P. 267,633).—See IV.

Pigment dyes (Dan. P. 34,077).—See IV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Changes in slab rubber on keeping. W. Spoon (Comm. Netherlands Rubber Testing Stat., Buitenzorg, 1927, No. 9, 67—73; Arch. Rubbercultuur, 1927, **11**, [2]).—Matured or slab rubber, if washed and crêped shortly after preparation, shows on storage a marked decrease in rate of vulcanisation, in tensile strength, and in viscosity. If stored in the original slab form the rate of vulcanisation and the tensile strength remain almost unchanged although the viscosity decreases. Considerable alteration, however, is evident in the condition of the surface rubber of the slabs, which becomes soft and finally tacky, and has a lower viscosity than the interior. D. F. Twiss.

Influence of "uspulun" on the inner properties of rubber. W. Spoon (Comm. Netherlands Rubber Testing Stat., Buitenzorg, 1927, No. 10, 74—78; Arch. Rubbercultuur, 1927, **11**, [2]).—Uspulun, a disinfectant containing a mercury compound of chlorophenol, has been recommended for application to the tapping surface of *Hevea* trees. The presence of 0·01% of uspulun in latex slightly retards coagulation, but has no serious effect on the inner properties or on the ageing behaviour of the rubber. D. F. Twiss.

Patents.

Manufacture of vulcanisation accelerators. Naugatuck Chemical Co., Assees. of S. M. Cadwell and A. T. Maximoff (E.P. 259,933, 13.9.26. Conv., 15.10.25) —Condensation of aldehydes, *e.g.*, heptaldehyde, with basic nitrogenous compounds, *e.g.*, aniline, is effected at temperatures above 100°, the optimum range being 135—145°. The products are much more active accelerators of vulcanisation than those obtained from the same materials by the earlier process (cf. E.P. 180,978; B., 1923, 899 a), in which the reagents are mixed at the ordinary temperature, and no external heating is applied. D. F. Twiss.

Vulcanisation of rubber. Rubber Service Laboratories Co., Assees. of C. O. North and C. W. Christensen (Can. P. 258,626, 14.5.25).—Vulcanisation of rubber is accelerated by the condensation product of an aromatic amine and an unsaturated aldehyde with conjugated double linkings. D. F. Twiss.

Vulcanisation of rubber. Goodyear Tire & Rubber Co., Assees. of C. E. Boord and E. N. C. Coolidge (Can. P. 260,248, 21.12.25).—The reaction product of 2-mercaptobenzthiazole and a guanidine compound, *e.g.*, a diarylguanidine, is applied as an accelerator of vulcanisation. D. F. Twiss.

XV.—LEATHER; GLUE.

Swedish Government regulations concerning the weighting of leather and methods of analysing it. E. Norlin (J. Soc. Leather Trades' Chem., 1926, **10**, 443—468).—Treatment of leather usually sold by weight with any substances not requisite for efficient tanning and finishing, or with excess of tanning materials for weighting purposes, is forbidden. Barium, magnesium, lead, tin, or other mineral salts, also glucose, molasses, dextrin, and similar substances are permitted only when used for bleaching or finishing. The water-soluble matter must not exceed 22·5% for butts, sides, etc., and 27·5% for cut bellies, neither must the ash exceed 3% for butts, sides, etc., nor 3·5% for cut bellies, the figures to be calculated in all cases on a basis of 18% moisture. Samples (each 10 × 10 cm.) shall be taken from each parcel of not more than 1000 pieces, selecting 3% of the pieces at random, but not less than 6. Details as to selection and cutting of samples are given. The finely-cut sample, reduced to 120 g. by repeated mixing and dividing, is bottled. *Moisture.*—Weigh 5 g. into a weighing bottle, dry for 3 hrs. at 100—103°, cool, and weigh. *Ash.*—The ash from 5 g. of the leather calcined in a weighed crucible, preferably in a muffle furnace at a dull red heat (550—600°), is cooled and weighed. *Fat.*—Extract 20 g. of leather in a paper thimble, the top of which is covered with filter paper, in a Soxhlet extractor, using light petroleum (b.p. 40—60°) and siphoning every 10 min. for 3 hrs. The solvent is finally distilled into the upper vessel, the contents of the extractor flask are distilled off, and the last traces of solvent are removed by means of a current of air. The residue is dried for 1 hr. at 100—103°, cooled, and weighed. The extracted leather is freed from light petroleum by heating it with hot water in a special distilling apparatus. The fat extraction is complete after 3 hrs. *Water-soluble matter.*—A Koch extractor is used, consisting of an extraction bottle securely closed with a rubber bung through which pass two glass tubes with funnel-shaped ends in the bottle covered with fine silk net. One tube terminates just inside the bung and acts as inlet tube from a flask containing water 1 m. above the extraction bottle. The other tube nearly touches the bottom of the bottle and acts as outlet. The fat-free leather is placed in the extraction bottle, the bung replaced, and the bottle filled with distilled water, shaken, left overnight, then placed in a double-walled water bath at 45°. The water in the flask automatically passes into the extraction bottle as the water extract passes out of the latter into a flask (1000 c.c.) which is filled in 3—3·5 hrs. The extract is cooled, made up to 1 litre, filtered through a Berkefeld filter candle, and 50 c.c. are evaporated to dryness and weighed. D. Woodroffe.

Chrome tanning. IV. Properties of basic chromium chloride liquors. E. Stiasny and D. Balányi (Collegium, 1927, 86—106. Cf. B., 1926, 376).—It is immaterial whether green or violet chromium chloride is used since the differences disappear when 33% basic liquors are aged. The addition of alkali to a solution of chromium chloride sufficient to make it 30% basic results in the formation of a precipitate of a hydroxochromium compound of the type

$$[(H_2O)_3(OH)Cr\langle{}^{OH}_{HO}\rangle Cr(OH)(OH_2)_3]Cl_2$$

which gradually dissolves again on ageing, heating, or drying owing to the formation of "ol" compounds of the type

$$[(H_2O)_4Cr\langle{}^{OH}_{HO}\rangle Cr(OH_2)_4]Cl_4$$

which can be made 70% basic before they precipitate.

The increased acidity of the "ol" compounds may be due to their greater hydrolysis, to the splitting off of acid, or to the disturbance of the hydrolysis equilibrium. When sodium hydroxide (1 mol.) is added to chromium chloride (1 mol.), hydroxopenta-aquochromium chloride is formed together with small amounts of the dihydroxo-salt and traces of chromium hydroxide. On being kept, the hydroxo compound and especially the dihydroxo-compound form "ol" compounds which increase the acidity and redissolve the precipitated chromium hydroxide, hence the liquor clears. Chromium complexes in which the "ol" formation has progressed most and which contain fewer hydroxyl groups per atom of chromium have the same basicity as the compounds in which "ol" formation has not taken place, but the basicity can be increased much more before they precipitate. If sodium carbonate solution is added to a chromium chloride solution, carbonato-chromium complexes are formed. Thus, a bluish-violet coloration is produced by adding sodium carbonate solution to blue hexa-aquochromium chloride or to green dichlorotetra-aquochromium chloride. After several weeks the colour changes to a deep green. The further addition of sodium carbonate gives green hydroxo- and "ol" compounds. Freshly tanned chrome leather is more susceptible to the action of acids or alkalis than aged leather. This is due to "ol" formation since tetrahydroxotetra-aquodiol-chromium is insoluble in acetic acid but soluble in hydrochloric acid, whereas polyol chromium is insoluble in both acids. The bluish-violet solution obtained by dissolving cold precipitated chromium hydroxide in hydrochloric acid is due to the formation of hexa-aquochromium trichloride; the hot precipitated compound contains "ol" groups which will not combine with hydrochloric acid; hence they give a green solution of octa-aquodioldichromium tetrachloride. The differences between the one- and two-bath chrome tannages are partly due to "ol" formation, which is much greater in the former process, hence a more astringent tannage is produced. One-bath chrome tanning liquors can vary according to chromium content, basicity, p_H value, precipitation figure, degree of masking, neutral salt content, and the degree of "ol" compound formation. As "ol" formation increases, the size of the chromium complexes grows and the pure crystalloid hydroxo-compounds become converted first into semicolloidal basic salts in which there is partial "ol" formation, and then into highly colloidal compounds in which the "ol" formation is complete. Neither the purely crystalloid salts nor the highly molecular compounds are of tanning value. D. WOODROFFE.

Possibility of use of sinew collagen for the determination of tannin in tanning extracts. W. S. SADIKOV (Collegium, 1927, 76—85).—Carefully separated cattle sinew was washed, finely divided, shaken three times for 6 hrs. each with 0·25% solution of caustic soda, washed with water and coagulated with acetic or carbonic acids, pressed into flakes, dehydrated with alcohol or acetone, and ground into a powdery mass. The product contained one third of the water-soluble matter of official hide powder. The solubility of the tanned sinew collagen was greater than that of the untanned, but practically equal to that of tanned hide powder. Hide powder was more absorbent than sinew collagen. Treatment with formaldehyde or basic chromium chloride strongly diminished its adsorbent properties. Comparative tannin analyses with hide powder and sinew collagen, respectively, made on several extracts showed close agreement. D. WOODROFFE.

XVI.—AGRICULTURE.

Action of potash fertilisers containing magnesium. E. HASELHOFF (Landw. Versuchs.-Stat., 1926, **105**, 75—104).—Field trials of the effect of various potash fertilisers with and without magnesium salts, on various crops, are described. On wheat and red clover magnesium salts produced an adverse effect on the crop yield, but on oats a somewhat larger crop was obtained. With a number of other crops the effect of magnesium salts was irregular, varying with the nature of potassium and magnesium salts used. Alterations in the composition of crops following the use of potash fertilisers was not great. The magnesium content of the crop varied least, even when magnesium salts were applied to the soil. Dressings of magnesium exerted a definite influence on the potassium content of the crop, but no regularity in its action was apparent. In pot experiments the influence of magnesium salts on the fertilising effect of potassium was variable, but the general tendency was to decrease yields. The sulphate, chloride, and carbonate of magnesium produced exactly similar results. No decided influence of magnesium on the intake of potassium by the plant was observed. A. G. POLLARD.

Influence of twelve years' continuous potash manuring on the crop yield, and the physics, chemistry, and mycology of the soil. H. NIKLAS, A. STROBEL, and K. SCHARRER (Landw. Versuchs-Stat., 1926, **105**, 105—136).—Crops benefitting most by potash manuring were potatoes, roots, and rye. Observed differences between soils continuously manured with potassium salts and unmanured controls continued to increase after twelve years' treatment. No appreciable effect on the reaction of the soil or of the plant sap followed the manuring with potassium salts, and the commonly observed physical properties remained unchanged. Definite but small and unimportant alterations in soil structure could be observed microscopically in the manured soils. Repeated heavy dressings of potassium fertilisers did not markedly reduce bacterial numbers in the soil, except in the case of potassium chloride, where a definite decrease in spores and bacterial numbers resulted. In general, no very unfavourable effects of continuous potash manuring were observed. A. G. POLLARD.

Significance of the occurrence of manganese, copper, zinc, nickel, and cobalt in Kentucky blue grass. J. S. McHARGUE (Ind. Eng. Chem., 1927, **19**, 274—276).—Kentucky blue glass (*Poa pratensis*, "English spear grass"), collected in the spring, was found to contain considerably more nitrogen, potassium and phosphorus, one-third as much calcium, appreciably more copper, and considerably more manganese and zinc than alfalfa. The association of high manganese and chlorophyll contents is significant, as previous work has shown the necessity of manganese for plant growth and chlorophyll synthesis. The beneficial effect of

manganese has been verified by growth experiments, and copper is also beneficial—a fact which may be associated with the presence of traces of copper in substances (including blue grass) containing the fat-soluble vitamin A, and may be of biological significance. As has been noted with other plants, the seeds of blue grass contain more copper, manganese, and zinc than the blades, these metals being concentrated in the germ and not in the endosperm. It is suggested that these metals are essential to the production of vitamins in plants.

C. O. HARVEY.

Growth of certain micro-organisms in planted and in unplanted soil. J. K. WILSON and T. L. LYON (Cornell Agric. Exp. Sta. Mem., 1926, No. **103**, 1—25).—Some crop plants (maize, timothy) produce a condition in the soil surrounding their roots (liberated organic matter) which favours the growth of certain micro-organisms (*Azotobacter*, *Bacillus radicicola*, *B. fluorescens*, a "nitrate-reducing organism," a "guttation organism" from maize, and a "blue organism" from soil) which consume nitrate-nitrogen, thus accounting for the disappearance of nitrate not absorbed by the plants.

CHEMICAL ABSTRACTS.

Comparative biological investigation of stable manure and "super" manure. G. RUSCHMANN (Z. Spiritusind., 1927, **50**, 15—16, 24—26, 35—36).—The "super" manure, produced by hot fermentation at 60—65° in a deep layer, is dark-brown to black, crumbly, practically odourless, and only faintly alkaline. It is distinguished from ordinary stable manure by its paucity in germs of every kind. High temperature and exclusion of air, which tend to diminish the growth of germs, are essential factors in its preparation. Although "super" manure produces the greater number of spores, its actual content is low compared with other manures owing to the extensive dying off which occurs. High-temperature fermentation also favours the formation of thermophile flora, and is attended by a loss of combined water and consequent enrichening in carbon. The exclusive use of the chemical evaluation of manures is often misleading, and should be supplemented by a biological examination.

F. R. ENNOS.

XVII.—SUGARS; STARCHES; GUMS.

Application of spectrometric measurements in the [beet] sugar industry. H. LUNDÉN (Z. Ver. deut. Zuckerind., 1926, 780—800).—Spectrometric measurements of products at different stages of manufacture or refining are recommended as a means of judging of the effect of clarification, of affining, of treatment with char or carbon, etc. Certain colour shades correspond to certain flavours. *E.g.*, an amethyst shade is generally accompanied by a very unpleasant taste (resembling beet juice), and methods removing this colour also eliminate the taste. A sugar having an amethyst shade develops a much worse flavour on heating, its sweetness being thus almost eliminated.

J. P. OGILVIE.

Adsorption of inorganic ions by "Carboraffin" and "Supra-Norit" in the filtration of thick-juice. J. FIŠER (Z. Zuckerind. Czechoslov., 1926, **51**, 49—55, 57—59).—Silica is adsorbed neither by "Carboraffin" nor by "Supra-Norit," but iron and aluminium of all the inorganic ions are most readily taken up, whilst "Carboraffin" is the better medium for calcium. On the other hand, with magnesium, "Supra-Norit" has the preference. Alkalis are little taken up by either, and the same holds for sulphate ions. Chlorine is more adsorbed by "Supra-Norit" than by the other carbon.

J. P. OGILVIE.

Boiling-up of thin-juice. V. MAYER (Z. Zuckerind. Czechoslov., 1926, **51**, 65—70, 73—80).—If during beet-sugar manufacture the juice is carbonated at ordinary temperature, and re-heated at 75°, no appreciable elimination of calcium carbonate occurs; but if the carbonation is performed at 75°, and if the juice is filtered at that temperature, a juice containing a much lower amount of lime thus results. In general, the boiling of carbonated juice in beet factories is insufficiently prolonged.

J. P. OGILVIE.

Determination of sugar in the beet. V. STANĚK and J. VONDRÁK (Z. Zuckerind. Czechoslov., 1926, **51**, 101—108, 113—121).—The volume occupied by the insoluble mark in the hot aqueous digestion method is found to average 1·56 c.c., instead of 0·6 c.c., at present assumed to be the correction for the normal sugar weight. This makes the juice volume to be 21·8, instead of 23·0 c.c.

J. P. OGILVIE.

Influence of filter-paper on the polarisation of sugar solutions. K. VNUK (Z. Zuckerind. Czechoslov., 1927, **51**, 125—128, 133—139).—If the paper used for filtering in the polarisation of sugars contains less or more than 7%, the filtrate is concentrated or diluted. Paper containing this amount of water can be obtained by storing it in a desiccator over a saturated aqueous solution of ammonium thiocyanate.

J. P. OGILVIE.

Coloration of sugar products. E. SAILLARD (Compt. rend., 1927, **184**, 291—293).—Samples of syrups, brown sugars from tail fractions, and molasses for the years 1921—1926 have been examined colorimetrically against a solution of iodine, the results obtained leading to the following conclusions:—(1) Coloration due to non-sugar organic matter in the syrup varies from year to year, the value sometimes being twice that for the previous year; (2) coloration of the molasses is about 10 times as great as that of the syrup, and 70% of that in the brown sugar.

J. W. BAKER.

XIX.—FOODS.

Detection of minute amounts of naphthalene in flour. A. K. EPSTEIN and B. R. HARRIS (Cereal Chem., 1926, **3**, 60—62).—50 g. of flour are treated with 100 c.c. of 10% sulphuric acid and steam distilled, 125 c.c. of distillate being collected. After extraction with 5 c.c. of chloroform, the extract is dried with calcium chloride and tested with anhydrous sublimed aluminium chloride, when the formation of a violet colour shows the presence of naphthalene. In this way 1—1·5 pts. of naphthalene per 100,000 pts. of flour may be detected. The test is more sensitive than that based on odour.

F. R. ENNOS.

Mechanical modification of dough to make it possible to bake bread with only the fermentation in the pan. C. O. SWANSON and E. B. WORKING (Cereal Chem., 1926, **3**, 65—83).—A machine is described for mixing dough with a pack–squeeze–pull–tear action. By mixing dough for 7 min. with the machine rotating at

120 r.p.m. a good quality bread may be obtained, without any fermentation other than that occurring during panning. If the mixing is excessive or is combined with the usual period of fermentation, the bread produced is poor in quality. F. R. ENNOS.

Rapid method for determining the gasoline colour value of flour and wheat. D. A. COLEMAN and A. CHRISTIE (Cereal Chem., 1926, 3, 84—89).—5 or 20 g. of flour or ground wheat are mechanically stirred in a closed bottle for 15 or 30 min. After filtering, the colour of the liquid is compared with a standard 0·005% potassium chromate solution in a suitable colorimeter. The colour values obtained are in substantial agreement with those determined by the lengthy A.O.A.C. process. F. R. ENNOS.

Gasoline colour value of several classes of wheat. D. A. COLEMAN and A. CHRISTIE (Cereal Chem., 1926, 3, 188—193).—Gasoline-soluble pigments of wheat and wheat flour vary greatly, and on the average the largest quantities were found in the hard red winter class of wheat, the common white class coming next, and the durum wheats having the least amount. Certain varieties of durum wheats (Acme, Monad, Pentad) have consistently less gasoline-soluble pigments than others (Arnautka, Kubanka, Kahla, and Mindum). No significant relationship was found to exist between colour and protein or ash content. D. G. HEWER.

Effect of monocalcium phosphate upon the viscosity of acidulated flour-in-water suspensions. L. EARLENBAUGH (Cereal Chem., 1926, 3, 102—106).—Addition of monocalcium phosphate to acid suspensions of flour in water diminishes the viscosity, but has no apparent effect on the quality factor. F. R. ENNOS.

Rapid colorimetric determination of hydrogen-ion concentration of crackers. R. T. BOHN and R. J. MARTZ (Cereal Chem., 1926, 3, 183—187).—The degree of alkalinity of biscuits may be determined by placing several drops of distilled water on the freshly-broken surface of the cracker and several drops of an aqueous or alcoholic solution of phenol red on this, and comparing the colour formed with those of standard tubes for this indicator. If the colour is near the limit of the range of the indicator cresol red is used. D. G. HEWER.

New factor for converting the percentage of nitrogen in wheat into that of protein. D. B. JONES (Cereal Chem., 1926, 3, 194—198).—The protein of the wheat endosperm consists chiefly of gliadin and glutenin containing, respectively, about 17·6% and 17·5% N, which gives a conversion factor of 5·7. The proteins of bran are essentially composed of an albumin (15·4% N), a globulin (17·7% N), and a prolamine (15·3% N), corresponding to a conversion factor of 6·31. The factor for the embryo is 5·8, based on a composition of 10% of albumin, 5% of globulin, and about 3% of proteose. Taking the whole wheat kernel as composed of endosperm 73·3%, bran 22·3%, and embryo 4·4%, the factor for the whole kernel is 5·83, compared with the usually adopted factor of 5·7. D. G. HEWER.

Test-bakes and certain of their chemical and physical aspects. F. L. DUNLAP (Cereal Chem., 1926, 3, 210—215).—The superiority of an actual baking test over analytical results as a measure of the quality of flour is emphasised. A standard technique for baking tests, however, is shown to be difficult to prescribe. Individual flours need characteristic treatment in mixing, kneading, fermenting, etc. in order to produce the best loaf. In the best-conditioned doughs the gluten is soft, pliable, and evenly distributed. Such conditions are favoured by a lower p_H value of the flour than obtains in many natural flours. A lowered p_H value and a short fermentation period tend to produce a well-built loaf, and in such cases the first of the two fermentation periods of the dough should be the shorter one. A. G. POLLARD.

Standardising the experimental baking test. M. J. BLISH (Cereal Chem., 1926, 3, 216—222).—The formulation of a standard baking test is rendered difficult by the different interpretations placed upon the terms "baking quality" and "baking strength." In actual practice the personal element in test-baking is considerable, and means for its elimination or minimising are needed. The question as to how far it is possible or advisable to standardise the details of baking-test technique is discussed. A. G. POLLARD.

Relation of crude protein content of flour to loaf volume. C. H. BAILEY and R. C. SHERWOOD (Cereal Chem., 1926, 3, 393—401).—A positive correlation has been found between the crude protein content of straight-grade flour milled from hard spring wheat and the volume of loaf produced from it. W. O. KERMACK.

Rapid moisture-testing oven for cereal chemistry laboratories. D. A. COLEMAN and H. B. DIXON (Cereal Chem., 1926, 3, 419—426).—A commercial oven for the determination in 15 min. of moisture in samples of flour etc. is described, and details are given of the method. Good agreement is found between the results obtained by it and by the routine method. W. O. KERMACK.

Quality of New Zealand wheats and flours. L. D. FOSTER (Trans. Proc. New Zealand Inst., 1926, 56, 738—743; Chem. Abstr., 1926, 20, 3518).—Relationships exist between the amounts of lime and magnesia and the protein content, the magnesia content and the ratio of wet to dry gluten, and the phosphate content and amount of ash, but not between the lime and magnesia contents and the baking value. A. A. ELDRIDGE.

Relation of kernel texture to the physical characteristics, milling and baking qualities, and chemical composition of wheat. J. H. SHOLLENBERGER and D. A. COLEMAN (U.S. Dep. Agric. Bull., 1926, [1420], 1—16; Chem. Abstr., 1926, 20, 3517).—The hard, mottled, and starchy types of kernels of hard red spring, hard red winter, and durum wheat were examined. The hard kernel was highest in density and flour yield, superior in water absorption, loaf weight, and crude protein content, and slightly higher in ash, crude fibre, and acidity. The starchy type was slightly superior in fat content and bran yield, but was generally inferior in milling and baking qualities. A. A. ELDRIDGE.

Determination of baking value of wheat by measure of specific energy of deformation of dough. M. CHOPIN (Cereal Chem., 1927, 4, 1—13).—

The rate at which dough rises in baking is chiefly dependent on its plastic properties, which may be determined by means of mechanical tests. The dough is mechanically admixed with 2% sodium chloride solution, and moulded into test pieces, which are immediately stretched in an extensimeter provided with a recording pressure gauge. Each piece is pressed into a disc of standard thickness and diameter, and expanded into a bubble by means of air pressure supplied by allowing water to flow into a graduated burette; the pressure within the bubble is recorded by the gauge. From the form of the curve traced by the gauge, and the volume of water admitted before the bubble bursts, the elastic properties can be computed; a suitable value for expressing the baking value is the specific energy of deformation, W. Variations in W are largely due to variations in the content and elastic properties of the gluten; the function W is approximately additive.

S. I. Levy.

Determination of iron, calcium, magnesium, phosphorus, ash, and protein in hard spring wheat and in the flour streams representing the largest volume of flour produced in its milling. E. P. Harding and G. Dysterheft (Cereal Chem., 1927, 4, 47—57).—The determinations were made on flours from a mill in Minneapolis, milling hard spring Marquis wheat from N. Dakota, and producing four break streams, four middlings, a straight flour, and a clear flour. All the components determined, except ash, are lower in the middlings than in the break streams, and lower in the straight than in the clear flour. The iron–ash and iron–magnesium ratios are almost constant throughout.

S. I. Levy.

Determination of amino-acids and proteolytic activity in wheat and flour. H. J. Denham and G. W. S. Blair (Cereal Chem., 1927, 4, 58—62).—The increase of amino-acids in the extract with the time during which the suspension is kept before centrifuging is not due to a gradual increase in the amount extracted (Swanson and Tague, B., 1916, 649; 1917, 469), but more probably to proteolytic activity in the flour, since it is observed if the suspension be immediately centrifuged, but is inhibited if the flour be first heated to destroy the enzymes. S. I. Levy.

Wheat and flour studies. IX. Density of wheat as influenced by freezing, stage of development, and moisture content. P. F. Sharp (Cereal Chem., 1927, 4, 14—46).—The density of wheat grown and dessicated under uniform conditions is approximately constant at about 1·48; it is not affected by light freezing, but is increased by severe freezing before maturity. Moisture content and moisture history affect the density more than other factors, but protein content and protein quality are also of importance.

S. I. Levy.

Effect of concentration on viscosity of flour suspensions. G. W. S. Blair, G. Watts, and H. J. Denham (Cereal Chem., 1927, 4, 63—67).—The results and theoretical considerations put forward by Sharp and Gortner (B., 1924, 29) are discussed. The straight line curve obtained by plotting the logarithm of the viscosity against concentration is suggested as of greater practical utility than the more complicated function plotted by those authors. S. I. Levy.

Increasing the protein content of potato waste. W. Ekhard (Z. Spiritusind., 1927, 50, 23).—A cattlefood with an increased protein content may be made by mixing potato waste with lupin seeds or with whey from cheese making. The composition of a number of such mixtures and their value as feeding stuffs are discussed, particularly in regard to the content of water and protein, and to the extent to which the bitter principle of the constituent lupin seeds has been removed, as is indicated by the modified Beckmann reaction. F. R. Ennos.

Composition of flaked maize. H. E. Woodman and J. Stewart (J. Agric. Sci., 1927, 17, 60—61; cf. B., 1925, 144).—Data are given for the composition of different commercial brands of flaked maize.

G. W. Robinson.

Chemical composition of manioc root. W. Ekhard (Z. Spiritusind., 1927, 50, 23).—A specimen of the whole root contained 13·07% of water, 1·46% of ash, was practically free from iron, and was distinctly alkaline. The water extract showed on analysis 13·24% of ash, 8·97% of protein, and 20·16% of reducing sugars as dextrose. On hydrolysis for $2\frac{1}{2}$ hrs. with dilute sulphuric acid, a further 14·40% of reducing sugar was obtained. F. R. Ennos.

Colorimetric determination of nitrogen in foodstuffs etc. W. Golub (Z. anal. Chem., 1927, 70, 119—128).—From 0·01 to 0·04 g. of the substance is heated with 2 c.c. of sulphuric acid (d 1·84) and a few drops of 33% perhydrol in a small test-tube until a clear, colourless solution is obtained, which, after cooling, is diluted to 100 c.c. 10 c.c. are titrated with sodium hydroxide, using phenolphthalein as indicator, and the same amount of alkali is added to a further 10 c.c., which are pipetted into a 100 c.c. graduated flask. After diluting to the mark, a quantity of the solution is analysed for ammonia by the Nessler colorimetric method. The mean of three tests should be taken. The results agree well with those obtained by the usual Kjeldahl process, but the analysis occupies only $1\frac{1}{4}$—$1\frac{1}{2}$ hrs. and requires no elaborate apparatus. Potassium chlorate may be used in place of perhydrol, but greater care is needed in the heating to avoid explosions. A. R. Powell.

Determination of sodium, potassium, and chlorine in foodstuffs. A. D. Husband and W. Godden (Analyst, 1927, 52, 72—75).—Sodium may be determined by ashing sufficient material to give a final weight of 0·1—10·15 g. of mixed sulphates, extracting the ash twice with hot N-hydrochloric acid, washing the residue with hot water, and making up filtrate and washings to 200 c.c. 100 c.c. of this extract are boiled, 5 c.c. of 10% barium chloride solution added, the mixture evaporated to about 25 c.c., made alkaline with ammonia solution, and then cooled. The precipitate is filtered off and washed with 2% ammonia solution, and the filtrate and washings are evaporated to dryness, after which 30—35 c.c. of alcoholic ammonium carbonate solution are added and the solution is made up to 100 c.c., and after 2 hrs. is filtered. 50 c.c. of filtrate

are evaporated to dryness, and the cold residue is treated with 5 c.c. of redistilled sulphuric acid, the excess of acid evaporated off, and the residue heated to dryness, the last traces of acid being removed by adding a little powdered ammonium carbonate before heating. After heating in the muffle the dish is cooled and weighed, and from the weight of mixed sulphates and the percentage of potassium the percentage of sodium can be calculated. Potassium is accurately determined by Green's method (cf. A., 1911, ii, 1135), and for chlorine care must be taken with the preliminary ashing to avoid loss. Various methods for chlorine determination have been examined and compared.
D. G. HEWER.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Ethyl phthalate. V. J. A. HANDY and L. F. HOYT (J. Amer. Pharm. Assoc., 1927, **16**, 7—17).—Esters of phthalic acid can be detected in essential oils by saponification with alcoholic potash and microscopical examination of the resulting potassium salt. The needle-like crystals of potassium phthalate can be readily distinguished from the potassium salts of other acids the esters of which are likely to occur in essential oils, with the exception of potassium carbonate, which, however, is easily recognised by other tests. The method is suitable for the gravimetric determination of ethyl phthalate, provided that other acids the potassium salts of which are insoluble in alcohol are absent. Saponification is best carried out at a comparatively low temperature (6—24 hrs. at 37°), and, since the crystals are very hygroscopic, they should be washed with absolute alcohol and filtered rapidly. A solubility correction should also be made. The results of analyses indicate that ethyl phthalate added to or present in various essential oils in amounts ranging from 1·38 to 16·5% can be recovered as the potassium salt practically quantitatively.
W. J. POWELL.

Manufacture of sodium salt of 3 : 3'-diamino-4 : 4'-dihydroxyarsenobenzene. P. A. KOBER (U.S.P. 1,611,461, 21.12.26. Appl., 12.4.20; cf. E.P. 24,152/1; B., 1915, 1165).—Arsphenamine base (3 : 3'-diamino-4 : 4'-dihydroxyarsenobenzene) is purified by precipitation with acid from an alkaline solution, and, after washing, is dissolved in an equivalent quantity of sodium hydroxide solution. On evaporation *in vacuo* at 50—80° the sodium salt of the arsphenamine base is obtained in a pure, stable form.
T. S. WHEELER.

Production of nuclear substituted aromatic cyano-mercury compounds. A. KLAGES (U.S.P. 1,618,095, 15.2.27. Appl., 1.9.21. Conv., 23.2.20).—See E.P. 188,376; B., 1923, 119 A.

PATENTS.

Preparation of hydrogenated polynuclear quinones. A. SKITA (F.P. 594,291, 2.3.25. Conv., 7.5.24 and 30.4.25; cf. A., 1926, 173).—Polynuclear quinones and their derivatives are catalytically hydrogenated, and the resulting nuclear-hydrogenated quinols (glycols) are oxidised.
C. HOLLINS.

Making nitroaryl alkyl ethers. L. S. PRATT, E. H. WELTZ, and W. L. MILLS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,619,368, 1.3.27. Appl., 18.10.20. Renewed 24.7.26).—In the preparation of nitroaryl alkyl ethers by heating together an alcohol, *e.g.*, ethyl alcohol, an alkaline hydroxide, *e.g.*, sodium hydroxide, and a halogenonitro-aromatic hydrocarbon, *e.g.*, *p*-chloronitrobenzene, side reactions due to the reduction of the nitro-group are inhibited by addition of an oxidising agent, *e.g.*, gaseous oxygen under pressure, or sodium peroxide.
T. S. WHEELER.

Production of a therapeutic substance from *Calotropis procera*. C. H. BOEHRINGER SOHN (G.P. 435,530, 8.4.24).—Aqueous extracts of *Calotropis procera*, with or without the addition of substances such as lead acetate to prevent the formation of emulsions, are treated with solvents, *e.g.*, chloroform, carbon tetrachloride, or trichloroethylene, and, by addition of ether or light petroleum to the solvent extract, a yellowish-white, micro-crystalline powder which has a cardiac action is precipitated.
E. H. SHARPLES.

Preparation of barbituric acid derivatives. F. HOFFMANN-LA ROCHE & Co., A.-G. (Swiss P. 113,251, 5.1.25).—Easily soluble alkylamine salts are formed by exposing 5 : 5-disubstituted barbituric acids in thin layers in a closed vessel containing a dish of anhydrous alkylamine, some arrangement for renewing the surface of the layer of acid being desirable. The *diethylamine* salts of diethyl-, phenylethyl-, ethylbutyl-, and *iso*-propylbromoallyl-barbituric acids, and the *dipropylamine* salt of *iso*propylallylbarbituric acid are described.
C. HOLLINS.

Extraction of essential oils from plants without distillation. E. FORAY (Addn. No. 30,574, 16.5.25, to F.P. 587,686).—Material containing essential oils is treated with the vapour of solvents such as ether, and solid material such as salt, sugar, sago, or flour, is added to the condensed vapour.
L. A. COLES.

Decomposition of glucosides [in plants prior to the extraction of perfumes]. E. FORAY (F.P. 609,548, 12.12.24).—Glucosides present in plants such as the iris or jasmine are hydrolysed by electrolytic treatment in the presence of water, prior to extraction of perfumes by the usual process.
L. A. COLES.

Extraction of perfumes from flowers. I. G. FARBENIND. A.-G. (F.P. 610,734, 8.2.26. Conv., 25.3.25).—Hot air or other non-reacting gas is passed successively over the flowers and over absorptive material such as active charcoal or silica gel to absorb the perfume, which is then recovered by extraction or distillation.
L. A. COLES.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Absorption of moisture of the air by anhydrous sodium salts used in photography. A. and L. LUMIÈRE and A. SEYEWETZ (Bull. Soc. Franç. Phot., 1926, [iii], **13**, 309—311).—Anhydrous sodium carbonate readily absorbs moisture from the air, tending eventually to a salt containing only 60% of the anhydrous substance, and approximating to the formula $Na_2CO_3,4H_2O$. Anhydrous sodium sulphite does not appreciably absorb moisture from the air. Anhydrous sodium thiosulphate readily absorbs moisture to form the stable salt $Na_2S_2O_3,5H_2O$.
W. CLARK.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 10, 1927

I.—GENERAL; PLANT; MACHINERY.

Kinetics of catalysed gas reactions in flow systems. A. F. Benton (Ind. Eng. Chem., 1927, **19**, 494—497).—It is assumed that the phenomenon of catalysis is a result of adsorption or of the formation of a superficial layer of an intermediate compound. The variation of adsorption with pressure is given by $s/S = ap/(1+ap)$, where s/S is the proportion of the area covered. When adsorption is small it is therefore proportional to the pressure. In the case of two reacting gases it will be proportional to the product of their partial pressures. If of two gases one is strongly adsorbed $dp/dt = k_1Sp_2 \cdot ap_1/(1+ap_1)$ and the partial pressure of the product is inversely proportional to the rate of passage of the mixture, or the yield is independent of this rate. The divergence from this result in the synthesis of ammonia is explained by adsorption of the product. From these considerations equations are deduced to cover special cases, the effective pressure of each gas being taken to be the mean of the pressures in the entering and exit gases. The catalysis of hydrogen and oxygen by silver is a case where oxygen and water are strongly adsorbed, but not hydrogen. Here it is shown that the yield should vary with the square root of the rate of flow. In the synthesis of ammonia, where neither of the reacting gases is strongly adsorbed, the equation is $xv = 3k_1Sp16ax—k_2S$, where x is the volume fraction of ammonia and v the rate of gas flow. In the case of the contact sulphuric acid reaction, all three gases are strongly adsorbed. In the conditions met with in practice the yield is $k_1S/aF—k_1S/2a$ where F is the fractional conversion of sulphur dioxide. Experimental results are in each case compared with these equations. It is shown that with uniform conditions over the cross-section, the shape of the catalyst mass is immaterial.

C. Irwin.

Condensation of a gas mixture to form an ideal solution. J. H. Simons (Ind. Eng. Chem., 1927, **19**, 482—485).—The quantity relations in the condensation of a gas mixture of three or more components can only be calculated on the assumption of an ideal solution. This assumption is nearly true for the liquefaction of air and for the condensation of members of a homologous series, *e.g.*, petrol vapour. A series of equations is developed by which, the vapour pressures of the pure components being known, the dew point and the point of total condensation may be calculated and the composition of vapour and liquid at these or intermediate temperatures ascertained. Composition curves based on calculation are given for the condensation of oxygen, nitrogen, and argon between 85·5° Abs. and 82·3° Abs., the dew point and point of total condensation at 1·395 atm., respectively. These show good general agreement with the results of experiment.

C. Irwin.

Automatic devices for the extraction of powdered materials. S. Palkin and H. R. Watkins (Ind. Eng. Chem., 1927, **19**, 535—537; cf. B., 1925, 566).—Two types of extractor are described in which the solvent flows upwards through the material to be extracted, thus maintaining it in a state of loose suspension and ensuring complete extraction. The first type consists of a boiling tube connected at the top to a reflux condenser, and having near its mid-point a side tube bent downwards to connect with a flask containing the solvent. The filtering medium, which may be absorbent cotton, is tightly sandwiched between the wall of the boiling tube and a cylinder of wire gauze, which latter has a folded groove running from the top to the point of entry of the side tube, to permit free passage of the solvent vapour to the reflux condenser. Down the centre of the boiling tube passes a narrow tube, closed at the lower end by a pad of absorbent cotton and funnel shaped at the top to receive the condensed solvent. The material to be extracted is situated in the annular space between the gauze and the inner tube. When in operation, the condensed solvent flows down the central tube, passes upwards through the solid material, and after filtering through the gauze and cotton, overflows into the heating vessel. In the second type the heating flask is provided with a long vertical tube sealed through the bottom of a wide boiling tube. Over the top of the inner tube is inverted a long test-tube, which rests loosely on the bottom of the boiling tube, in which is contained the powdered material suspended in solvent. Solvent vapour from the flask passes upwards through the inner tube, and, escaping from the bottom of the test-tube, maintains the solid in constant agitation. The filtering and overflow devices are substantially the same as in the first type, and the whole extraction vessel is water-jacketed. A liquid seal inside the heating flask prevents vapour entering the return pipe.

H. J. Dowden.

Continuous extraction apparatus. A. H. Clark (Ind. Eng. Chem., 1927, **19**, 534—535).—The apparatus described is of the Soxhlet type, capable of dealing with large quantities of material. The container consists of a pharmaceutical percolator, the bottom of which is closed by a cork and connected to the flask containing the solvent through a straight tube provided with a stop-cock, and having a vertically-directed insertion below the cock. (Alternatively, this side tube may be replaced by a suitably bent tube passing into the solvent flask).

The top of the percolator is closed by a cork or clamped wooden disc and washer, and is connected to the usual reflux condenser through a straight tube having a side tube branching downwards. The two side tubes are connected by rubber tubing or a Liebig condenser, which for less volatile solvents, *e.g.*, alcohol, is used as a steam jacket to prevent undue condensation. When in operation the outflow from the percolator is regulated by the stop-cock to equal the rate of condensation in the reflux. The extraction then becomes continuous, there is no siphoning, with its inherent irregularities, involved, and the operation can be stopped at any moment by closing the stop-cock. H. J. DOWDEN.

Lubricating oils. Laboratory tests in relation to practical results. A. G. MARSHALL and C. H. BARTON (J.S.C.I., 1927, **46**, 130—138 T).—The validity of a number of laboratory tests usually applied to lubricating oils was examined by means of engine trials. It was found that oiliness had no practical significance in the lubrication of internal-combustion engines. Oxidation and coking tests of the types at present in use were found to give results which enormously exaggerated the difference between different lubricating oils in those cases in which they were in line with practical results, and in many cases they were found to be blended with residual filtered cylinder stock, with the result of "improving" their tests from the popular specification point of view. Such blending, however, increases the danger due to deposits in service. Evaluation of an oil by means of the rate of change of viscosity with temperature was also found to be misleading.

Glass wool as insulator. BATES.—See VIII.

PATENTS.

Method of furnacing in thermo-chemical treatment. C. B. JACOBS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,625,610, 19.4.27. Appl., 24.2.21).—Charges which shrink during thermo-chemical synthetic reactions are treated until a substantial shrinkage occurs, a further charge is then added before the completion of the original batch, and the augmented batch is treated to completion. W. G. CAREY.

Apparatus for producing refrigeration. Storing device for gas and liquids. F. G. KEYES, Assr. to NATIONAL REFRIGERATING Co. (U.S.P. 1,622,519—1,622,523, 29.3.27. Appl. [A—E], 19.2.20, 11.5.20, 8.6.21, 6.11.23, and 4.12.23).—In a refrigerating system comprising a still, a condenser, and refrigerator proper, with a cooling means used alternately for the still and the condenser, electrical means are used to effect the change over and cut off the heat from the still, which may be controlled by the level of liquid in the condenser or in the refrigerator. Check valves may be used in the pipe connexions between the still and the condenser, and between the refrigerator and still, whilst a capillary tube may be used between the condenser and refrigerator so that liquid may continually pass to the refrigerator while the still is working. In (E) a form of electrically heated still is described. B. M. VENABLES.

Heating granular materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 268,599, 21.6.26).—Granular materials such as contact masses, or sulphur and granulated coal for the production of carbon disulphide, are heated uniformly in an electric furnace by forcing gases through them to maintain constant agitation. Steam superheated at 250° thus agitates coke granulated between 1—10 mm. in a furnace heated electrically by steel electrodes, and water-gas is produced continuously. W. G. CAREY.

Apparatus for detecting and determining impurities and dissolved matter in water and other fluids. EVERSHED & VIGNOLES, LTD., and C. E. PERRY (E.P. 268,597, 18.6.26).—In apparatus, such as the "Dionic Water Tester" described in E.P. 23,706 of 1907 and 12,735 of 1914 (cf. B., 1908, 1036; 1915, 454), in which the impurities and dissolved matter contained in water are ascertained from a measurement of the electrical conductivity of the water, the temperature correction is effected by varying the effective electrical length of the column of liquid being tested, a temperature scale being provided to enable this length to be readily adjusted. J. S. G. THOMAS.

Separation of constituents of ternary gaseous mixtures. W. WILKINSON, Assr. to AIR REDUCTION Co., INC. (U.S.P. 1,619,909, 8.3.27. Appl., 15.10.25).—A primary liquefaction and rectification separates a liquid rich in the least volatile constituent and a gas rich in the most volatile constituent. Intermediate vapours are withdrawn to another rectifier not in heat communication with the primary one, but the gaseous product of the auxiliary is liquefied by heat exchange with a cold product of the primary rectifier and returned to the auxiliary rectifier. The liquid product from the auxiliary is returned to the primary rectifier. B. M. VENABLES.

Gas analysis apparatus. R. W. JAMES. From BROWN INSTRUMENT Co. (E.P. 268,637, 3.9.26).—In gas analysis apparatus of the catharometer type in which the relative thermal conductivities of two gases or gas mixtures are compared electrically, the cells in which the gases are respectively received are formed in a single metallic block, *e.g.*, of aluminium, which may be coated with wax or varnish, the block being contained in a metallic housing provided with gas passages, so that temperature differences in the two gases are minimised. The gas passages are provided with filters for cleaning and drying the gases during passage to the respective cells. J. S. G. THOMAS.

Centrifugal separator. T. C. THOMSEN, Assr. to KOEFOED, HAUBERG, MARSTRAND, & HELWEG AKTIESELSKABET TITAN (U.S.P. 1,625,919, 26.4.27. Appl., 9.7.26. Conv., 8.3.26).—See E.P. 259,514; B., 1926, 999.

Separating gaseous mixtures. J. LE ROUGE, Assr. to SOC. L'AIR LIQUIDE (SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (U.S.P. 1,626,345, 26.4.27. Appl., 1.2.23. Conv., 16.3.22).—See E.P. 195,046; B., 1923, 638 A.

[Heating chamber] for gas- or oil-fired furnaces. R. F. HISLOP (E.P. 268,421, 29.12.25).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Composition of coal. Resolution of coal by solvents. C. Cockram and R. V. Wheeler (J.C.S., 1927, 700—718).—The γ-compounds of bituminous coals (compounds soluble in pyridine and chloroform—6·9% of coal), on extraction with (*a*) light petroleum, (*b*) ether, and (*c*) acetone, are separated into the following percentage fractions: (*a*) 33, (*b*) 37, (*c*) 12, and (*d*) residue, 18. From a study of the destructive distillation, oxidation with hydrogen peroxide and nitric acid, and ultimate analysis the compositions of the fractions are (*a*) saturated hydrocarbons, 40%; unsaturated hydrocarbons, 40%; resins, 20%; (*b*) resinols, resines, and resenes; (*c*) and (*d*) resin-like compounds. The treatment of bituminous coal with benzene under pressure at 200° gave an extract amounting to 5·96% of the coal. Fractionation of this extract with the above solvents gave products similar to those obtained by the pyridine-chloroform extraction. At 285° the extract amounted to 7·3% of the coal, but the general characters of the resulting fractions were the same. Tables showing the results obtained from different coals are given.

H. Burton.

Differentiation of peat and lignite. U. Springer and G. Abele (Brennstoff-Chem., 1927, **8**, 117—120).—Differentiation along geological, botanical, or petrographical lines is unreliable, as is also the physical method of determining the adsorptive power towards a dilute solution of ferric chloride, which varies considerably with the acidity and the fineness of the specimen. The degree of decomposition of the cellulose in the material may be measured by determining the degree of hydrolysis produced by 72% sulphuric acid, but the values found for different types of coal often lie very close together. A simpler and more reliable differentiation is afforded by the pentosan content of the material, which is very rarely less than 2% for peat or greater than 1% for lignite, though even so, intermediate types are inevitably met with.

W. T. K. Braunholtz.

Path of travel of the gases in the coke oven. II. and III. W. E. Davies (Gas World, 1927, 86, Coking Sect., 2—5, 12—16; cf. B., 1927, 4).—A further discussion of the influence of the plastic layer resistance and the variation of the gas pressure in the hot and cool zones on the flow of gases in the coke oven. The rate of heating of successive layers of material across the oven and the amount of fissuring occurring in the coke layer are important factors in determining the pressure gradients. The work of Foxwell and others is criticised in the light of the author's theory of the mechanism of the coking process. A number of applications of the theory to coke-oven practice are summarised.

A. B. Manning.

Reactivity of coke. G. Agde and H. Schmitt (Brennstoff-Chem., 1927, **8**, 121—123).—The differences in reactivity exhibited by different cokes are attributed to somewhat other factors than those described by Nettlenbusch (cf. B., 1927, 208). The reactivity of a coke depends essentially on the accessibility of its carbon, particularly its amorphous carbon, to carbon dioxide, this being determined by the specific number, form, and diameter of those pores of which the surface is accessible to the gas without unduly prolonged diffusion. The two principal factors in deciding this are the nature of the parent coal and the degree of decomposition of the tar coke (the solid decomposition product of non-expelled tar) corresponding to the carbonising temperature. Whilst graphite may be deposited on coke through decomposition of methane, it is also derived from decomposition of the tar coke at high temperatures. The reactivity of coke, under given experimental conditions, may vary as the experiment proceeds, owing either to partial gasification of the accessible carbon, or to an increase in the quantity of accessible carbon through changes in shape (*e.g.*, due to fracturing).

W. T. K. Braunholtz.

Determination of relative ignitabilities and combustibilities of domestic cokes. Tests on the possibilities of a "brazier and weighing method." T. F. E. Rhead and R. E. Jefferson (J.S.C.I., 1927, **46**, 166—172 T).—A method for determining the relative ignitability and combustibility of cokes for domestic use, by the continuous weighing of a coke fire in a brazier, has been partially investigated and some of the difficulties have been overcome. The apparatus used is briefly described, and results are given showing the effects of grade of coke, size of fire, and grate area on the rate of combustion.

Determination of the phenol content of gas liquor [crude ammonia liquor] and effluents from coke-oven by-product plants [and gasworks etc.]. H. Bach and H. Uthe (Brennstoff-Chem., 1927, **8**, 120—121).—Existing volumetric and gravimetric methods of determining phenol in crude ammonia liquor are briefly discussed. An improved method, in which all sulphur compounds which are oxidised by bromide are completely removed, is to acidify the liquor (100 c.c.) and distil twice to very small bulk, filter the phenolic distillate, and boil gently under a reflux condenser until all hydrogen sulphide has been expelled (about ¼ hr.). About 1 c.c. of aqueous barium chloride and two drops of phenolphthalein are added, and then dilute sodium hydroxide in slight excess. The solution is again boiled to aggregate the precipitated barium sulphate and carbonate, and filtered, the phenol in the filtrate being determined with bromine by Koppeschaar's method.

W. T. K. Braunholtz.

Vaporisation of petroleum. E. H. Leslie and A. J. Good (Ind. Eng. Chem., 1927, **19**, 453—460).—The vaporisation of a paraffin-base petroleum has been studied in an apparatus consisting of a heating coil and an equilibrium chamber in series, immersed in a constant-temperature bath. The liquid was fed at a constant rate of 10 c.c./min. into the heating coil and, on attaining equilibrium, the liquid and vapour phases were collected separately and analysed by a method giving their true b.p. curves. Equilibrium "single-flash" vaporisation of the petroleum was carried out by this method at 250°, 300°, 350°, etc., up to 600° F. The vapours all contained high-boiling compounds and the residues low-boiling compounds. The proportion of the distillate, *i.e.*, the condensed vapour, which boiled below the flash temperature was approximately constant

and averaged 82·3%. In a second series of experiments the crude oil was first vaporised at 250°, the residue then vaporised at 300°, and so on up to 600° F.; this constitutes "successive-flash" vaporisation. It is less efficient than "single-flash" vaporisation in yield and in composition of the distillate, and also in heat requirement. The proportion of distillate boiling below the flash temperature was 80·61%. The results are discussed with regard to their application to petroleum distillation practice. A. B. MANNING.

Petroleum lubricants. C. F. MABERY (Ind. Eng. Chem., 1927, **19**, 526—529; cf. B., 1926, 810).—Determinations have been made of the sp. gr., viscosity, proportion distilling below 300° (30 mm.), and the sp. gr. and viscosity of the distillate and residue, of a number of lubricants from American petroleums. The behaviour of the original lubricant, of the distillate below 300°, and of the residue, in a Carpenter friction-testing machine, has also been studied. In some instances the distillate supports a heavier load than the original, and in others it breaks under a lighter load; the residues show similar variations in stability. The residual hydrocarbons obtained after extraction of the residue above 300° with an alcohol-ether solvent approximate to the series C_nH_{2n-8} for the Pennsylvania oils, and to C_nH_{2n-10} and C_nH_{2n-12} for the others. Two oils subjected to heavy use have been examined and found to have undergone little deterioration. There appears to be no direct relation between the viscosity and the stability under load, oils of the same viscosity showing wide variations in frictional tests. A series of oils of the same viscosity at 38° (320 sec.) showed a maximum difference in viscosity of 12 sec. at 98° and of 27 sec. at 54·4°.

A. B. MANNING.

Action of "antiknocks." A. EGERTON and S. F. GATES (Nature, 1927, **119**, 427).—Antiknocks do not influence detonation in rapid combustion mixtures; they function in the initial stages of the combustion as negative catalysts. Those metals which give effective organo-metallic antiknocks are capable of forming higher oxides. Only those types of purely organic substances are effective as antiknocks which retard the oxidation, at the ordinary temperature, of certain liquid aldehydes. A. A. ELDRIDGE.

Determination of [hard] asphaltum content of mineral oils. J. MARCUSSON (Chem.-Ztg., 1927, **51**, 190).—The methods used hitherto for the determination of asphaltum are neither concordant nor accurate. It is now proposed to use an ethereal solution of ferric chloride as the precipitant. 5 g. of oil are dissolved in 50 c.c. of ethyl ether and 5 c.c. of a 5% ethereal solution of ferric chloride added. The precipitate is filtered off and washed with ether, extracted with boiling ether, and dissolved in warm chloroform. This solution is agitated first with 5 c.c. of dilute hydrochloric acid, then with 5 c.c. of water, and finally, after evaporation to dryness at 105°, the residue is weighed.

H. MOORE.

Valuation of commercial motor spirits by Ostwald's index number method. W. OSTWALD (Petroleum, 1927, **23**, 445—448).—The author traverses the criticisms raised by Kroch (B., 1927, 272), and maintains that the method gives reliable commercial results, even with benzine-benzol mixtures. It is of use as indicating the amount of b.p. depression in alcohol mixtures. H. MOORE.

Powdered coal for kiln firing. HARTFORD.—See VIII.

Linings for water-gas manufacture. BRADY.—See VIII.

PATENTS.

Coking retort oven. J. VAN ACKEREN, Assr. to KOPPERS CO. (U.S.P. 1,613,068, 4.1.27. Appl., 14.2.21).—Each of the oven walls contains a series of up-flow and a series of down-flow flues which can be interconnected. Two channels communicate with each series of combustion flues, the channels being alternately used to supply fuel gas and air to the up-burning flues, and to receive the waste gases from the down-burning flues. S. PEXTON.

Process for briquetting coal. S. R. WAGEL, Assr. to LEHIGH COAL & NAVIGATION CO. (U.S.P. 1,618,029, 15.2.27. Appl., 30.9.24).—Coal is bonded with clay, bitumen, and a binding agent which is effective between the temperature at which bitumen loses its binding qualities and that at which the clay becomes sufficiently baked to consolidate the whole. S. PEXTON.

Binder for briquetting or moulding fuels, minerals, earths, and other finely-divided substances. A. TAPPING (E.P. 263,942, 6.10.25).—An aqueous pulp of starch containing alkali or sodium silicate and a preservative, *e.g.*, cresol or formaldehyde, is mixed with pitch or bitumen of fairly high m.p. in a heated chamber under pressure. The temperature is maintained above the m.p. of the bitumen and the mixture is stirred. The moisture content of the product is regulated, after mixing, by allowing the escape of steam therefrom. The product may be used either in the liquid form, or cooled and the solid binder mixed and ground with the materials to be bonded.

S. PEXTON.

Manufacture of useful products by oxidising coal. W. A. BONE and R. QUARENDON, Assrs. to GAS LIGHT & COKE CO. (E.P. 268,006, 14.12.25).—Coal, or the residue obtained after heating coal under pressure with benzene, is oxidised, preferably at raised temperature, with a solution of caustic alkali, and an alkali manganate or permanganate, at ordinary pressure or in an autoclave. After filtration, the alkali salts of benzenoid carboxylic acids, *e.g.*, mellitic acid, are separated.

W. G. CAREY.

Manufacture of liquid fuels. I. G. FARBENIND. A.-G. (E.P. 252,018, 10.5.26. Conv., 8.5.25. Addn. to E.P. 226,731; B., 1925, 163).—In liquid fuels containing iron carbonyl, the quantity of the latter is reduced by adding organic compounds of metals or metalloids soluble in benzene, *e.g.*, methyl or ethyl borate or silicate, silicon or lead tetraethyl, zinc oleate, etc. The fuel may also contain organic halogen compounds such as ethylene chloride, chloroform, carbon tetrachloride, and/or organic compounds containing oxygen or nitrogen (cf. E.P. 252,019; B., 1927, 357). W. G. CAREY.

Manufacture of combustible gas. W. D. WILCOX (U.S.P. 1,624,644, 12.4.27. Appl., 1.4.21).—A mixture

of coal- and water-gas is generated by passing steam through coal (contained in two generators) previously raised to incandescence by means of an air blast. The water-gas produced is superheated by passage through regenerators (previously heated by the combustion of the blast gases) and then passes upwards through a charge of fuel, having a layer of uncarbonised coal, to a gas outlet. C. O. Harvey.

Treatment of gas containing carbon monoxide. W. H. Kniskern, Assr. to Atmospheric Nitrogen Corp. (U.S.P. 1,614,072, 11.1.27. Appl., 21.9.23).—Gas to be purified from carbon monoxide is brought into contact with a catalyst in the presence of steam. The heat of treated gases is passed on to the incoming untreated gases, which are then brought into contact with circulating water, producing steam for the reaction. S. Pexton.

Desulphurising coal, water, or mixed gases for illuminating or heating. E. Raffloer, Assr. to W. E. Leuchtenberg (U.S.P. 1,624,625, 12.4.27. Appl., 3.1.25).—The gas is blown into a desulphurising chamber along with finely-divided material capable of absorbing sulphur. C. O. Harvey.

Apparatus for the destructive distillation of oil shale. L. R. Abernathy (U.S.P. 1,618,038, 15.2.27. Appl., 1.12.24).—Oil shale is distilled in superheated steam in a retort, dome shaped at the top. Within the dome is a conical hood from the underside of which the products of distillation are withdrawn. The raw shale entering the retort falls on to the outside of the hood, where it is preheated and distributed in the retort. S. Pexton.

Decolorising and clarifying agent [for petroleum]. H. S. Christopher, Assr. to Standard Oil Co. of California (U.S.P. 1,617,476, 15.2.27. Appl., 30.9.22).—Pure hydrated aluminium silicate having the approximate composition $Al_2O_3,12SiO_2,2H_2O$ is claimed. S. Pexton.

Apparatus for distilling hydrocarbons under pressure and a catalysing agent. F. M. Hess (U.S.P. 1,625,467, 19.4.27. Appl., 29.9.22).—The hydrocarbons to be cracked are vaporised in a still and the vapours passed through a primary dephlegmator and thence to a secondary dephlegmator into which a catalysing agent is introduced. C. O. Harvey.

Extraction of bituminous materials. I. G. Farbenind. A.-G., Assees. of E. Reissmann and A. Richter (G.P. 437,010, 8.11.25).—The fraction of mineral oils, tars, etc. which is soluble in liquid sulphur dioxide is used as an extracting agent; higher yields are obtained than when benzene is used. A. B. Manning.

Manufacture of an emulsion. D. Finley, Assr. to Paraffine Cos., Inc. (U.S.P. 1,625,304, 19.4.27. Appl., 17.1.23).—Hot bitumen is added slowly with agitation to lime slaked with water, with which is mixed a hot solution of alum, the temperature being maintained. H. Royal-Dawson.

Treatment of emulsions of hydrocarbon oils and water. Standard Development Co. From C. F. Pester (Can. P. 247,810, 25.6.24).—The emulsions are treated with $\frac{1}{4}$—5% of the sludge obtained in refining mineral lubricating oils with fuming or concentrated sulphuric acid, and the mixture is heated at about 77°. A. B. Manning.

Iron carbonyl preparation. Badische Anilin- u. Soda-Fabr. From A. Mittasch and M. Müller-Cunradi (Can. P. 262,601, 11.7.25).—Iron carbonyl is dissolved in a hydrocarbon, the solution containing at least 20% of the former. Hydrocarbon substitution products which are inert to iron carbonyl can also be used as solvents. A. B. Manning.

Conversion of petroleum hydrocarbons. R. Cross, Assr. to Gasoline Products Co. (U.S.P. 1,624,778, 12.4.27. Appl., 29.10.23).—The oil accumulates in a pool in an initial stage, and passes thence through a heating stage to a converter which has open vapour communication with the initial stage, whereby the evolved vapours pass through the relatively cooler oil and thence to the dephlegmator and condenser. The unvaporised oil provides energy for producing a turbulent condition in the converter and maintaining the carbon in suspension. C. O. Harvey.

Preparation and treatment of olefines. Petroleum Chemical Corp., Assees. of H. S. Davis and W. J. Murray (E.P. 248,375, 24.2.26. Conv., 24.2.25).—The olefine mixtures resulting from the cracking of petroleum vapours at a nearly constant temperature between 600° and 650° are subjected to a physical fractionation in suitable plant of which a diagram and detailed description are given. Three main fractions are collected containing: (1) ethylene and propylene, (2) Δ^{α}- and Δ^{β}-butylenes and *iso*butylene, and (3) Δ^{α}- and Δ^{β}-amylenes, *iso*propylethylene, *as*-methylethylethylene, and trimethylethylene, together with hexylenes and higher olefines. The fractions are then selectively absorbed in diluted sulphuric acids of various strengths or in hydrochloric acid, which effects a further separation of their constituents owing to their varying reactivity with acid. The resulting alkylsulphuric acids or alkyl chlorides, on hydrolysis, yield the corresponding alcohols. *E.g.*, from the cracking of "gas oil" (d 0·856) at 601° there are obtained per barrel (42 gals.) 2·6 gals. of the amylene fraction. This is agitated with 12·2 lb. of 65% sulphuric acid at below 20° for 6 hrs., allowed to settle, and the lower acid layer separated, neutralised, and distilled, when 0·28 gal. of crude tertiary alcohols (chiefly amyl) is obtained, resulting from the interaction of trimethylethylene, methylethylethylene, and some higher olefines with the acid, and subsequent hydrolysis. The upper layer of residual olefines, containing the amylenes and *iso*propylethylene, is distilled up to 60°, yielding 1·1 gals. of distillate. This is treated with 15·3 lb. of 77% sulphuric acid at 35—40° for 6 hrs., when the above olefines are absorbed. After settling, the lower acid layer is diluted with water so as to reduce the acid strength below 20%, and distilled to obtain 0·56 gal. of crude secondary amyl alcohols. Similarly, the butylene fraction (102 cub. ft.) obtained from the gas oil is treated with 65% sulphuric acid, which extracts *iso*butylene etc., and yields, on hydrolysis, 0·28 gal. of tertiary butyl alcohols. The residue, on treatment with 77% sulphuric acid, produces on hydrolysis 0·46 gal. of secondary alcohols formed from the butylenes. A. Davidson.

Pulverised fuel burners. H. A. PROCTER (E.P. 268,417, 30.1.26).

Apparatus for the generation of gas from liquid hydrocarbons for combustion, lighting, and other purposes. R. E. GOLDSBROUGH, and GOLDSBROUGH PATENTS Co., LTD. (E.P. 266,765, 1.9.25).

Heating granular materials (E.P. 268,599).—See I.

Gas analysis apparatus (E.P. 268,637).—See I.

Material similar to compressed asphalt (E.P. 260,621).—See IX.

Bituminous emulsions (E.P. 268,411).—See IX.

Organic substances from gas mixtures (E.P. 262,404).—See XX.

III.—TAR AND TAR PRODUCTS.

Determination of phenol. BACH and UTHE.—See II.

PATENTS.

Separation of the components of low-temperature tar without distillation. ZECHE M. STINNES, and A. WEINDEL (G.P. 433,455, 5.9.23).—The crude tar is treated with benzine at the ordinary temperature, and, after separation of the precipitated pitch and removal of the benzine by distillation, the residual oil is resolved into neutral oil and phenols by treatment with alcohol of less than 60% strength. The use of caustic soda being avoided and the solvents used being recoverable, the process is economical. A. B. MANNING.

Separation of the phenols and neutral oils of low-temperature tar. ZECHE M. STINNES, and A. WEINDEL (G.P. 436,444, 22.7.22. Addn. to G.P. 433,268). —The procedure of the principal patent is modified by the use of a mixture of alcohol and a concentrated solution of ammonia, the mixture containing not more than 60% of alcohol for the extraction of the phenols. The solubility of the phenols in alcohol is increased several times by the addition of ammonia, so that extraction is facilitated, and the phenols are recovered directly in a highly pure form. Thus 32% of phenols, purity 98—99%, were recovered by one extraction from a low-temperature tar of which the total phenol content was 40%. The ammonia and alcohol are recovered by distillation without appreciable loss. A. B. MANNING.

Asphalt substitute. J. DRESCHER (Austr. P. 104,402, 4.12.23).—Lignite tar, heated if necessary, is mixed with sulphur, resin, and a mineral filler such as sand or basalt, the mixture brought to 80°, and treated with powdered lime. A. B. MANNING.

Distillation of tar and the like. W. B. DAVIDSON, A. C. MICHIE, and E. W. MUDDIMAN (U.S.P. 1,626,588, 26.4.27. Appl., 23.4.26. Conv., 1.4.25).—See E.P. 255,919; B., 1926, 815.

Bituminous emulsions (E.P. 268,411).—See IX.

IV.—DYESTUFFS AND INTERMEDIATES.

Food dye. H. JOHNSON and P. STAUB (Ind. Eng. Chem., 1927, 19, 497—498).—The green dye known as Fast Green F.C.F., now proposed for admission to the list of permitted colours, is formed by the condensation of 2 mols. of ethylbenzylanilinesulphonic acid with 1 mol. of *p*-hydroxybenzaldehyde-*o*-sulphonic acid followed by oxidation with lead peroxide. The *p*-hydroxybenzaldehydesulphonic acid is prepared from *p*-nitrotoluene-*o*-sulphonic acid by oxidation in alkaline hypochlorite solution to the corresponding stilbene compound, which is converted into *p*-nitrobenzaldehyde-*o*-sulphonic acid by oxidation with alkaline permanganate. The *p*-nitro-compound is reduced with ferrous carbonate, diazotised, and warmed at 80—90° with dilute sulphuric acid, whereby *p*-hydroxybenzaldehyde-*o*-sulphonic acid results. The dye is superior, both as regards fastness and intensity, to the dyes at present in use. H. J. DOWDEN.

PATENTS.

Preparation of azo dyes. I. G. FARBENIND. A.-G. (F.P. 611,004, 12.2.26. Conv., 13.2.25).—Azo dyes for acetate silk and wool are obtained by coupling diazotised toluidine-ω-sulphonic acids with aromatic amines which contain no sulphonic or carboxylic groups. Examples are: *p*-toluidine-ω-sulphonic acid → diphenylamine (yellow), 5-nitro-*o*-toluidine-ω-sulphonic acid → benzylethylaniline (red), 3 : 5-dinitro-*o*-toluidine-ω-sulphonic acid → ethyl-β-naphthylamine (blue-violet). The dinitro-compound is obtained by amination of 2-chloro-3 : 5-dinitrotoluene-ω-sulphonic acid. C. HOLLINS.

Preparation of [azo] dyes containing chromium. GES. FÜR CHEM. IND. IN BASLE (F.P. 609,518, 19.1.26. Conv., 16.2.25).—Azo dyes containing chelate groups are heated with a solution of chromic chromate. Examples are: 4-chloroaniline-3-sulphonic acid → salicyclic acid (greenish-yellow on wool); 4-chloro-2-aminophenol-5-sulphonic acid → 1-phenyl-3-methyl-5-pyrazolone (red); 1-amino-2-naphthol-4-sulphonic acid → 1-*m*-nitrophenyl-3-methyl-5-pyrazolone (bluish-red). C. HOLLINS.

Preparation of vat dyes of the anthracene series containing nitrogen. I. G. FARBENIND. A.-G., Assees. of M. KUNZ (G.P. 436,537, 17.2.25).—Amino- and diamino-anthraquinones or their arylidene derivatives, and substitution products of these types, are condensed with aldehydoanthraquinones or their derivatives to give vat dyes. Vat dyes are thus obtained from 2-aminoanthraquinone and 2-aldehydoanthraquinone (orange-yellow) or 1-chloro-2-aldehydoanthraquinone (yellow) or 1-amino-2-aldehydoanthraquinone (claret); from 1-chloro-2-aminoanthraquinone and 1-amino-2-aldehydoanthraquinone (claret-red) or 1-azido-2-aldehydoanthraquinone (salmon-pink *product*, m.p. 331—333°); from 1-chloro-2-benzylideneaminoanthraquinone and 2-aldehydoanthraquinone (orange-yellow); from 2 : 6-dibenzylidenediaminoanthraquinone and 2-aldehydoanthraquinone (yellow), or 1-amino-2-aldehydoanthraquinone (claret *product*, m.p. over 350°), or 1-chloro-2-aldehydoanthraquinone (yellow *product*, m.p. over 360°); and from 2-benzylideneamino-3-methylanthraquinone and 1-amino-2-aldehydoanthraquinone (pink *product*, m.p. above 340°). C. HOLLINS.

Preparation of condensation products of the anthraquinone series [*iso*dibenzanthrones]. I. G. FARBENIND. A.-G., Assees. of A. WOLFRAM (G.P. 436,077, 12.8.24. Addn. to G.P. 426,710. Cf. E.P. 255,277; B.,

1926, 780).—3 : 9-Diaroylperylenes, heated with aluminium chloride at high temperatures, are converted into *iso*dibenzanthrones. C. Hollins.

Preparation of acid wool dyes of the anthraquinone series. I. G. Farbenind. A.-G., Assees. of W. Mieg (G.P. 436,539, 15.11.24).—The sulphonated (acridinated) products from dianthraquinonylamines are reduced, *e.g.*, with sodium hydrogen sulphide, probably to dihydroacridines with elimination of one sulphonic group. 1 : 1′-Dianthraquinonylamine, after disulphonation with 15% oleum at 95—100°, is reduced by sodium hydrogen sulphide to a *compound*, which in the presence of bisulphite dyes wool an olive-green, becoming brown on after-chroming. Di-α-anthraquinonyl-1 : 5-diaminoanthraquinone yields a similar dye. From 1 : 2-dianthraquinonylamine by treatment with 15% oleum and subsequent reduction with alkaline sodium sulphide there is obtained a red acid *dye*; reduction with copper dust and sulphuric acid in place of sodium sulphide leads to a yellow-brown acid *dye*, giving brown shades on after-chroming. Di-α-anthraquinonyl-2 : 6-diaminoanthraquinone, from 2 : 6-dichloroanthraquinone and 1-aminoanthraquinone, gives, by treatment with 10% oleum at 95—100° followed by reduction with alkaline hyposulphite, a brownish-red acid *dye*. C. Hollins.

Manufacture of vat dyestuffs. M. P. Schmidt, Assr. to Grasselli Dyestuff Corp. (U.S.P. 1,625,826, 26.4.27. Appl., 20.11.23. Conv., 25.11.22).—See E.P. 207,553; B., 1924, 1008.

Manufacture of dyestuffs containing chromium. F. Straub, Assr. to Soc. Chem. Ind. in Basle (U.S.P. 1,626,167—9, 26.4.27. Appl., 29.1.26. Conv., [A], 14.2.25, [B, C], 16.2.25).—See E.P. 247,556; B., 1926, 910, and F.P. 609,518, preceding.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Constituents of cell-wall of flax fibre. A. E. Cashmore (J.C.S., 1927, 718—731).—The "cellulose-complex" obtained from flax fibre by freeing the fibre from extraneous matter, from pectin by ammonium oxalate treatment, and from hemicellulose by washing with 4% aqueous sodium hydroxide, had an α-cellulose content of 82—83%. Hydrolysis of the cellulose-complex with 2% barium hydroxide solution at 100° gave galactose together with small amounts of pentoses. With 5% sulphuric acid at 100° practically no hydrolysis took place, but at 130—140° dextrose, galactose, fucose, xylose, and traces of "uronic acid" were produced. Part of the dextrose produced is obtained by the hydrolysis of the non-cellulose portion of the complex. Water at 140—150° hydrolyses the complex yielding a hemicellulose (3·5% of weight of fibre) containing about 21% of "uronic anhydride," probably mainly galacturonic anhydride, 60% of galactose, and 10—14% of rhamnose. In addition, there is obtained a syrup soluble in 70% alcohol (1% of weight of fibre) containing xylose and fucose. At temperatures above 175° water disintegrates the fibre. H. Burton.

Determination of cellulose by saccharification. A. Kiesel and N. Semiganovsky (Ber., 1927, 60, [*B*], 333—338).—Dry cellulose is preserved with 7—10 times its amount of 80% sulphuric acid for 2½ hrs. at the atmospheric temperature. Water is added in the proportion of 15 c.c. for each c.c. of acid used, and the resulting solution is heated for 5 hrs. on the steam bath. Cellulose is thereby converted quantitatively into dextrose. The presence of proteins (gelatin and casein) is without influence on the change. Dextrose, mannose, galactose, lævulose, invert sugar, xylose, and arabinose retained 99·7, 97·7, 99·9, 26·0, 66·06, 72·2, and 84·5% of their reducing power after treatment with acid as above. For the determination of cellulose in botanical material, the substance is subjected to a preliminary treatment with 2% hydrochloric acid for 3—5 hrs. on the water bath, and saccharification of the residue is accomplished as described above, or, alternatively, the process may be applied to the "crude fibre" obtained by one of the customary methods. Direct saccharification leads to correct results only in the absence of ketoses and pentoses. H. Wren.

Sulphite boiling process. M. Hönig and W. Fuchs (Ber., 1927, 60, 782—786).—A very marked increase is observed between the consumption of iodine by original sulphite liquors and those which have been preserved in about 1% alkaline solution. The amount of loosely combined sulphur dioxide is greater than that which can be accounted for by the aldehydes and sugars present. Further, the amount of organically combined sulphurous acid in an original sulphite liquor is identical with that of a "fermented" liquor. The sulphur dioxide must therefore be considered to be combined with the ligninsulphonic acid. The presence of an aldehydic group in the latter is improbable since the copper number of carefully dialysed ligninsulphonic acid is negligible. It is probable that the sulphurous acid is united to tautomerically active phenols. H. Wren.

Unsaturated sugar complexes in wood. W. Fuchs (Ber., 1927, 60, 776—782).—The yield of sugars obtainable by the hydrolysis of wood with acids can be considerably increased at the expense of the lignin by suitable pre-treatment of the material with perbenzoic acid, particularly if this has been preceded by the action of 0·5% sulphuric acid at the atmospheric temperature. The increase is due to the more copious presence of fermentable sugars, presumably dextrose, in the solutions from the oxidised wood. The pentosans in the original wood are decomposed to a considerable extent under the conditions of the hydrolysis, and the pentosan content of all the solutions from the wood subjected to the varied treatments is small and almost equal. The observations lend support to the hypothesis of the presence of unsaturated sugar complexes in wood. H. Wren.

Action of sodium hydroxide on cellulose under high pressure. S. Odén and S. Lindberg (Ind. Eng. Chem., 1927, 19, 132—133).—Cotton cellulose was treated with about 7 times its weight of approximately 3·5*N*-caustic soda solution and heated in an autoclave slowly to 372° and maintained at this temperature for some hours. After removal of gaseous reaction products the residual transparent yellow-brown solution was fractionated to recover methyl alcohol, acetone, and light oil, evaporated to dryness, and extracted with ether to

remove pitch. The residue, consisting of alkali salts of organic acids, was then steam-distilled at 110—585°, and the distillate fractionated under reduced pressure into light and heavy oils and pitchy matter. The reaction products resulting from these operations are tabulated and their quantities recalculated on the basis of 100 g. of pure ash-free cellulose. Ultimate analysis of these products reveals losses equivalent to about 3·25% of the original cellulose. The densities and refractive indexes of the oil fractions are recorded; these oils are highly unsaturated, and are possibly ketonic in character.

D. J. Norman.

Determination of soda. Textor and Hoffman.—See VII.

Use for hydrocellulose. Gardner.—See XIII.

Cellulose ester varnishes. Dabisch.—See XIII.

Patents.

Production of stiff fabrics which stand washing. A.-G. Cilander (E.P. 264,783, 1.3.26. Conv., 21.1.26).—Artificial silk is woven with vegetable or animal fibres and the resulting fabric exposed for a short time, *e.g.*, 15 secs., at about 15° to the action of an inorganic acid, *e.g.*, sulphuric acid (*d* 1·53). The strength of the acid, the duration of the treatment, and the proportion of artificial silk in the fabric determine the degree of stiffness.

D. J. Norman.

Manufacture of fibre board. R. W. Hilton, Assr. to Kemper-Thomas Co. (U.S.P. 1,624,599, 12.4.27. Appl., 1.11.23).—Weather-resistant fibre board is made by thoroughly mixing fibre pulp in an aqueous solution of soap and adding lead acetate to precipitate insoluble lead soap, the amount of soap being sufficient to provide not over 3% of insoluble soap in the fibre. W. G. Carey.

Manufacture of strawboard. M. E. Pennington and A. B. Davis (U.S.P. 1,625,090, 19.4.27. Appl., 18.4.25).—The strawboard comprises disintegrated new straw which has been disinfected and steam distilled, rendering it inodorous and sterile.

H. Royal-Dawson.

Waterproof and like materials. C. Häfele (E.P. 266,813, 3.12.25).—Two or more webs of fibrous material are compounded by means of a sulphur-free rubber solution containing approximately 10% and 5%, respectively, of finely-powdered zinc oxide and barium sulphate. The composite web is then satinised and dried by passing it between rollers at 140—160°. D. J. Norman.

Protection of animal fibres against attack by alkaline liquids. M. Bergmann (G.P. 437,836, 14.10.23).—Sulphite-cellulose waste liquors in which the active groups have been oxidised, reduced, or halogenated, or the active constituents of the liquor after they have been subjected to one of these processes, are used for protecting the fibres during treatment with alkaline liquids.

L. A. Coles.

Manufacture and use of artificial silk and the like. British Enka Artificial Silk Co., Ltd., Assees. of N. V. Nederlandsche Kunstzijdefabr. (E.P. 244,496, 15.12.25. Conv., 15.12.24).—The affinity for dyestuffs of artificial silk consisting of or containing cellulose silk, *e.g.*, viscose, may be increased by treatment with an oxidising agent, to increase the content of oxycellulose, or decreased by treatment with liquids which, whilst having a solvent action on some or all of the non-cellulose constituents, have no appreciable action on cellulose. For example, the affinity for dyestuffs of viscose may be increased by exposure for about 2 hrs. at the ordinary temperature to a solution of sodium hypochlorite containing 5% of available chlorine, or decreased by heating for about 24 hrs. at 150° with glycerin. D. J. Norman.

Manufacture of artificial threads or filaments. Soc. pour la Fabr. de la Soie "Rhodiaseta" (E.P. 259,190, 31.8.26. Conv., 1.10.25. Addn. to E.P. 233,384; B., 1925, 587).—In a modified process for the dry spinning of artificial threads involving the use of temperature-controlling elements, as described in the prior patent, the controlling elements are connected in parallel, and in order to obtain different temperatures in different parts of a cell more than one element may be used, the temperature-controlling fluid to one of them being cooled if necessary. B. P. Ridge.

Treatment of cellulose acetate or products made therewith. British Celanese, Ltd., and G. H. Ellis (E.P. 266,777, 29.10.25).—Pattern or other effects are produced on textile fabrics made from or containing cellulose acetate threads or yarns by the local application (by printing, stencilling, etc.) to the fabric of one or more substances which modify the delustring action of hot or boiling aqueous media or moist steam, and afterwards subjecting the material to such delustring treatment, preferably with moist steam. The applied substances may be mechanical resists, solvents for cellulose acetates, or substances soluble in cellulose acetate, those of the last two groups which are not sufficiently soluble in water being either dissolved in liquids which are non-solvents for cellulose acetate, or dispersed colloidally in aqueous media. The delustring may be prevented, retarded, or accelerated by the agents used, and the effects may be further enhanced by dyeing the fabric with suitable dyestuffs. B. P. Ridge.

Non-inflammable plastic masses. Pathé Cinéma, anc. Etabl. Pathé Frères (F.P. 612,414, 30.6.25).—Cellulose esters or ethers are mixed with the phosphoric esters of halogen-substituted aliphatic alcohols and the usual plasticisers and highly chlorinated compounds. *E.g.*, 1 kg. of nitrocellulose is mixed with 400 g. of tritrichloroethyl phosphate and 400 g. of a plasticiser (tricresyl phosphate etc.), and the whole dissolved in a volatile liquid. The mixtures serve for the production of artificial fibres, plastic masses, films, etc.

B. Fullman.

Manufacture of coated laid paper. G. W. Johnson. From A. M. Collins Manufacturing Co. (E.P. 267,013, 13.9.26).—Ordinary paper is coated preferably on both sides with, *e.g.*, a solution of casein or glue, optionally in admixture with fillers and colouring agents, and is then passed between embossing rollers which impress a laid pattern on the coating. The lines thus formed are blended with the main coating while this is still moist in order to give the effect of a watermark in the body paper. The sheet may, if desired, receive a second plain coating, and is finally rolled to produce an even smooth surface suitable for high-grade printing. D. J. Norman.

Manufacture of a waterproof [paper] composition. L. Kirschbraun (U.S.P. 1,621,791, 22.3.27. Appl., 20.10.20. Renewed 8.10.26).—Molten asphalt mixed with oleic acid is emulsified by agitation with water containing colloidal clay, and the emulsion is incorporated with a suitable paper or other fibrous stock, and treated with a substance, *e.g.*, lime, which precipitates oleic acid as an insoluble oleate. The treated stock is then passed through a papering machine and dried. T. S. Wheeler.

Manufacture of artificial silk and the like from viscose solutions. W. P. Dreaper (U.S.P. 1,626,454, 26.4.27. Appl., 18.4.25. Conv., 2.5.24).—See E.P. 239,254; B., 1925, 915.

Solvents for cellulose ester (E.P. 255,406).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Mordanting and dyeing. V. [Mechanism of the weighting of silk.] E. Elöd, L. Teichmann, and E. Pieper (Z. angew. Chem., 1927, **40**, 262—264).—The isoelectric point of a Japanese silk in the raw state was p_H 5·1, and after weighting with tin silicophosphate, 4·1. A similar displacement of this point is obtained by prolonged treatment first in hydrochloric acid then in sodium hydroxide solutions equipotential with the solutions used in the commercial weighting process. These results appear to confirm Elöd's theory of the mechanism of this process (B., 1924, 939; 1926, 316). A. R. Powell.

Action of iron blacks on leather. Woodroffe and Dew.—See XV.

Patents.

Dyeing wool and silk. I. G. Farbenind. A.-G. (F.P. 610,308, 1.2.26. Conv., 7.2.25).—Even shades on wool and silk mixtures are obtained by dyeing with azo dyes derived from pyrazolones and containing in either component one or more carbalkoxy-groups. C. Hollins.

Dyeing leather with acid azo dyes. I. G. Farbenind. A.-G. (F.P. 609,904, 23.1.26. Conv., 3.2.25).—Brown shades on leather are produced by using dyes of the type: amine→resorcinol←aminonaphtholsulphonic acid, or amine→resorcinol←aminonaphtholsulphonic acid←amine. In place of resorcinol an unsulphonated derivative may be used, and in place of the aminonaphtholsulphonic acid a napthylaminesulphonic acid. C. Hollins.

Dyeing and printing of cellulose esters. I. G. Farbenind. A.-G. (F.P. 610,539, 3.2.26. Conv., 6.3.25).—Sulphamic acids derived from coloured amines, especially from aminoanthraquinones, are dyed on cellulose acetate silk from an acid bath containing sodium sulphate. The sulphamic acid from 1:4-diaminoanthraquinone gives a violet-red, that from 4:8-diaminoanthrarufin a reddish-blue. For printing, the sodium sulphamate is mixed with glycerol and a thickener. C. Hollins.

Calico printing process. I. G. Farbenind. A.-G., Assees. of E. Münch and K. H. Meyer (G.P. 435,092, 10.2.25. Addn. to G.P. 433,153; B., 1927, 216).—The hydroxymethylformamide of the earlier patent is replaced in part or entirely by formamide. Formamide is a good solvent for dyes, especially for basic dyes, and this property is enhanced by addition of resorcinol. The mixture enables basic dyes to be fixed by steaming without addition of tannin or other fixing medium. C. Hollins.

Pattern printing on cloth with the help of wax. F. Adler (Austr. P. 104,395, 9.10.25. Conv., 7.11.24 and 13.7.25).—The material to be printed is laid on a flat bed of wax and then printed with a heated metal templet carrying the pattern, so that the wax under the printed portions melts and penetrates the material. The wax may be moulded on a cylinder and the pattern carried on a cylindrical printing roller. A waxed cloth of suitable length may be used in place of solid wax. The material to be printed should be stretched at a short distance above the wax bed so that after printing it frees itself from the reserving layer and adhesion is prevented. C. Hollins.

Preparation of padding-baths. I. G. Farbenind. A.-G. (F.P. 610,519, 2.2.26. Conv., 3.2.25).—Aromatic or partly hydrogenated aromatic sulphonic acids or their alkyl, aralkyl, or alkylaralkyl derivatives, or salts of these, are added to padding baths containing aniline salt, β-naphthol, 2:3-hydroxynaphthoic arylamides, etc. in order to increase the penetrating power of the solution, the resulting dyeings being thus rendered faster to washing and rubbing. Suitable sulphonic acids are those derived from benzylated naphthalenes, or the condensation of naphthalenesulphonic acids with *cyclo*hexanol, benzyl alcohol or chloride, or tetralin dichloride, or of tetralinsulphonic acid with *cyclo*hexanol. C. Hollins.

Dyeing [acetylcelluloses]. P. Rabe, Assr. to Grasselli Dyestuff Corp. (U.S.P. 1,622,122, 22.3.27. Appl., 11.4.24. Conv., 9.5.23).—See E.P. 215,783; B., 1925, 801.

Bleached and dyed furs (E.P. 264,969).—See XV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Solubility of calcium phosphates in citric acid. J. Graftiau (Bull. Soc. chim. Belg., 1927, **36**, 165—171).—Experiments in which a number of naturally occurring calcium phosphates were treated with a 2% solution of citric acid, initially in strict accordance with the requirements of the Wagner method, the treatment being then repeated on the extracted material till dissolution was complete, show that under these conditions the whole of the phosphoric acid is finally brought into solution. The rate of dissolution is markedly influenced by the state of aggregation. In general, the argillaceous phosphates of Liège and limestone phosphates require 4—5 successive extractions. Florida phosphates, land pebble, and hard rock, are much more resistant, and 8—11 successive extractions are necessary for the complete dissolution of the phosphoric acid. In the case of limestone phosphates application of the Wagner method leads to results which are difficult to interpret, since a not inconsiderable portion of the citric acid is immediately

neutralised by the calcium carbonate present. A specimen of limestone phosphate which yielded only 13·5% of its total phosphate content on a first treatment with a 2% solution of citric acid, yielded 40·8% and 31·1%, respectively, during the second and third treatments.

J. S. CARTER.

Determination of soda by electrical conductivity. C. K. TEXTOR and W. F. HOFFMAN (Paper Trade J., 1927, **84**, 201—205).—The method may be applied to cooking liquor, black liquor, washed pulp, and leached black ash. Although it is empirical, there is a definite relation between the total soda content and the specific conductivity of the liquors and effluents under the uniform conditions prevailing in any given mill.

CHEMICAL ABSTRACTS.

Potash from greensand. II. Adsorption from the vapour phase by glaucosil. C. W. WHITTAKER and E. J. FOX (Ind. Eng. Chem., 1927, **19**, 467—469).—Glaucosil is the siliceous residue left after the extraction of greensand with acid. It is grey in colour, white after ignition, and is more active chemically than crystalline silica. Adsorptions of benzene, xylene, water vapour, and carbon tetrachloride were determined from the pure vapours, and the total percentages by weight taken up were found to be 40·5, 31·0, and 61·0, respectively, for the three organic vapours mentioned. Water is also strongly adsorbed, and can be driven off only by heating at 200—250°. Adsorption–time curves are given showing a gradual fall from 100% to zero. In some cases initial low efficiencies were found when starting the apparatus, ascribed to a phase of preliminary activation.

C. IRWIN.

Determination of thiocyanates in the presence of chlorides, sulphides, and cyanides. Z. CHARATZ (Chem.-Ztg., 1927, **51**, 251).—The crude potash of beet vinasses often contains potassium thiocyanate (Italian samples up to 1% or more), which may be thus determined: 20 g. of the potash are dissolved in water, hydrochloric acid is added to neutrality or feeble acidity, the liquid boiled, and diluted to 200 c.c. In each of two identical 25 c.c. vessels are placed 0·5 c.c. of 0·4% ferric sulphate solution and 1 c.c. of 1 : 2 hydrochloric acid. The first is then filled with water, and the second treated with 5 c.c. of the test solution and filled with water. The blank is then titrated with 0·5% potassium thiocyanate solution until the colours are identical. 0·04% of thiocyanate in the crude substance may be thus determined. The presence of more than 0·1% of ferrocyanide is disturbing, but the difficulty is avoided by working at high dilution; chlorides and sulphides have no effect. Using ammonium thiocyanate, the thiocyanate in ammonium sulphate may be thus determined. Cyanides are determined after conversion into thiocyanate by treatment with ammonium sulphide.

B. FULLMAN.

Volumetric determination of carbon dioxide in carbonates. J. LINDNER and F. HERNLER (Z. angew. Chem., 1927, **40**, 462—464).—The material is decomposed with 4—10% hydrochloric acid in a slow current of air free from carbon dioxide, and the issuing gases are passed through an absorption tube in which is placed a definite volume of 0·1N-barium hydroxide containing 1% of barium chloride and 0·3 c.c. of a 0·1% solution of phenolphthalein as indicator. Without filtering off the precipitated barium carbonate the excess of barium hydroxide is titrated with 0·1N-hydrochloric acid containing 3% of barium chloride. A compact apparatus for carrying out the determination without exposure of the solutions to the atmosphere is illustrated diagrammatically.

A. R. POWELL.

Ratio of sulphur trioxide to phosphoric anhydride [in acid phosphate]. J. B. THOMAS and C. C. HOWES (Amer. Fertiliser, 1926, **65**, [8], 28—30).—The most economical ratio is determined by consideration of the cost of the acid in relation to the curve obtained by plotting the ratio of sulphur trioxide to phosphoric anhydride against the percentage availability of the phosphate. The method is not applicable in the manufacture of acid phosphate from rock, which differs greatly in composition from that used in establishing the ratio.

CHEMICAL ABSTRACTS.

Kinetics of catalysed gas reactions. BENTON.—See I.

Detection of mercury vapour. NORDLANDER.—See X.

Cyanides in the blast-furnace. KINNEY and GUERNSEY.—See X.

Liquids for mineralogical analyses. SULLIVAN.—See X.

Determination of phosphoric acid in fertilisers. BORDEIANU.—See XVI.

PATENTS.

Production of concentrated solutions of alkali cyanides. DEUTSCHE GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESSLER, and K. ANDRICH (E.P. 268,420, 23.12.25).—Hydrocyanic acid gas is absorbed, preferably below 15°, in an aqueous mixture of alkali sulphate and lime, starting with a quantity of alkali sulphate insufficient to cause precipitation of a double salt of alkali sulphate and calcium sulphate (in the case of sodium sulphate not more than 28%), and continuing the admission of hydrocyanic acid until some calcium cyanide is formed, which is transformed into alkali cyanide by the addition of more alkali sulphate. Excess of alkali sulphate is avoided by adding it in portions, at intervals, so that calcium cyanide is formed in the intervals.

W. G. CAREY.

Manufacture of potassium nitrate. A. LAMBERT (E.P. 267,996, 18.11.25).—Sylvite, carnallite, kainite, or other material containing potassium chloride is partially purified from sodium chloride by concentrating a solution, which is then mixed with a solution of caliche similarly purified; the mixture is further concentrated by evaporation to separate further sodium chloride, and the potassium nitrate formed by the double decomposition is crystallised.

W. G. CAREY.

Manufacture of ammonium nitrate in water solution and simultaneous concentration thereof. Rapid evaporation to dryness of ammonium nitrate solutions. AZOGENO S.-A. PER LA FABR. DELL'AMMONIACA SINTETICA E PROD. DERIVATI, and

C. TONIOLO (E.P. 247,227—8, 8.2.26. Conv., 7.2.25).—(A) Nitric acid diluted with ammonium nitrate solution is neutralised with gaseous ammonia mixed with air heated at 80—100°. The heat developed by the reaction is thus utilised to evaporate some of the water contained in the acid. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 6048 of 1915 and 11,731 of 1888.] (B) Molten ammonium nitrate, still containing some water (10—20%), is sprayed over a mass of pulverised, cold or hot ammonium nitrate, and a current of cold or hot air is introduced at the particular time at which the molten salt incorporates itself with the pulverised salt. W. G. CAREY.

Preparation of an alkaline, iron-treated carbon suitable for cyanide synthesis. J. MICHAEL & Co. (G.P. 435,975, 6.3.24)—Porous carbon is impregnated with a solution containing an alkali carbonate and a complex iron salt (sodium ferrocyanide, sodium iron tartrate, etc.) which gives no precipitate with alkali carbonate. Good yields of cyanide are claimed, using smaller proportions of iron than usual. C. HOLLINS.

Decreasing the rate of crystallisation of borax from brine. A. W. GAUGER and H. H. STORCH, Assrs. to BURNHAM CHEMICAL Co. (U.S.P. 1,621,927, 22.3.27. Appl., 29.8.25).—The crystallisation of borax during the evaporation of brine is inhibited by the addition of 0·05—0·1% of rosin, or of the extract obtained by treating coniferous sawdust or the bush *Larrea mexicana* with sodium carbonate solution. T. S. WHEELER.

Manufacture of rouge. P. J. HESS, Assr. to PITTSBURGH PLATE GLASS Co. (U.S.P. 1,618,086, 15.2.27. Appl., 1.2.21).—Ferrous sulphate is heated at 870—940° to drive off the water of crystallisation and to convert it into a compound consisting of approximately 2 pts. by wt. of ferric oxide to 1 pt. of ferric sulphate. This compound is mixed with water to form a polishing mixture. H. ROYAL-DAWSON.

Production of oxides. F. THARALDSEN (Nor. P. 40,618, 29.1.23).—Material to be oxidised is heated below its fusion point under oxidising conditions, mixed with a flux and reducing agents and fused, and then the oxidation is completed. L. A. COLES.

Manufacture of lead nitrate. W. H. BENTLEY, B. CATLOW, and W. BLYTHE & Co., LTD. (E.P. 268,104, 24.2.26).—Metallic lead is alternately lowered into and raised out of a lead nitrate solution, the moist lead being oxidised by a current of air and the film of oxide dissolved by the solution. Nitric acid is added to the liquor as required, keeping it neutral or slightly basic, and solid lead nitrate is obtained by crystallisation, after slightly acidifying a portion of the liquor. W. G. CAREY.

Regeneration of catalysts used in the production of phosphorus pentoxide or phosphoric acid by the interaction of phosphorus or phosphoretted hydrogen and water vapour. I. G. FARBENIND. A.-G. (E.P. 259,201, 20.9.26. Conv., 29.9.25).—The catalysts are regenerated in the reaction vessel by suspending the inflow of raw material and passing hydrogen containing a small amount of oxygen through the heated vessel. W. G. CAREY.

Production of hydrogen. E. EDWIN (F.P. 612,238, 8.10.25. Conv., 17.10.24).—Gases containing carbon dioxide, obtained in the reduction of iron oxide formed by treating iron with steam for the production of hydrogen, are reduced by passage through a high-tension electric arc and returned to the process. L. A. COLES.

Manufacture of moulded sulphur. RHENANIA VEREIN CHEM. FABR. A.-G. (E.P. 265,540, 30.6.26. Conv., 3.2.26).—Moulds of aluminium or its alloys are employed and are cooled with water or air. The length joints are provided with flanges so arranged that the plastic or elastic filling between them does not come into contact with the molten sulphur. W. G. CAREY.

Causticising units or apparatus. W. D. MOUNT (E.P. 265,669, 12.8.25).—See F.P. 608,190 ; B., 1927, 187.

Gas analysis apparatus (E.P. 268,637).—See I.

Heating granular materials (E.P. 268,599).—See I.

Hydrogen and oxygen (E.P. 268,426).—See XI.

VIII.—GLASS ; CERAMICS.

Glass annealing. J. W. FRENCH (J. Soc. Glass Tech., 1927, **11**, 10—19).—Conditions of importance in annealing are (1) heat emissivity of the surface at all temperatures, (2) thermal expansion, (3) thermal conductivity, (4) cohesion, (5) viscosity, (6) surface layer, (7) homogeneity, (8) form and dimensions. The problem, in general, does not lend itself to mathematical treatment. Much evidence supports the theory that glass is always coated with an amorphous surface layer with properties differing from those of the underlying material, and the layer yields with great readiness to small impact forces. The annealing process is discussed and two types of lehr designed for annealing optical glass are described. A. COUSEN.

Stress systems and photo-elastic phenomena [in glass]. F. W. PRESTON (J. Soc. Glass Tech., 1927, **11**, 23—29).—Photo-elastic phenomena can only be used successfully in studying the stress system in glass when restricted to two-dimensional cases, namely, by using plane parallel laminæ with stresses confined to the plane of the laminæ. With equal stresses at right angles, the ordinary polariscope indicates no strain, and, for accuracy, an instrument showing change of absolute refractive index parallel to each principal stress is needed. A. COUSEN.

Economics of the [glass] annealing process. E. A. COAD-PRYOR (J. Soc. Glass Tech., 1927, **11**, 30—36).—The temperature of containers straight from machines on entering the lehr ranged from 580° to 778°. An insulated lehr was designed such that the heat carried in by the ware was practically sufficient to maintain the annealing temperature. For safety, a small amount of additional heat was supplied by preheating the slats of the belt. A. COUSEN.

Investigation of the glassy state by the method of enforced crystallisation. J. F. PONOMAREV (J. Soc. Glass Tech., 1927, **11**, 39—52 ; cf. B., 1926, 877).—The m.p. diagrams of the following systems were determined by the method of enforced crystallisation : (1) $Na_2O,2B_2O_3$–B_2O_3, (2) $Na_2O,2B_2O_3$–$3CaO,P_2O_5$, (3) $Na_2O,2B_2O_3$–Al_2O_3, (4) $Na_2O,2B_2O_3$–MgO, (5) Na_2O,

$2B_2O_3$–CaO, (6) $Na_2O,2B_2O_3$–ZnO, (7) $Na_2O,2B_2O_3$–PbO. From its physico-chemical characteristics glass is defined as a strongly under-cooled fusion which, on crystallising from its molten mass, gives crystals of the same chemical composition as the mass itself.

A. COUSEN.

Transparent zirconia-containing glasses. V. DIMBLEBY, S. ENGLISH, E. M. FIRTH, F. W. HODKIN, and W. E. S. TURNER (J. Soc. Glass Tech., 1927, **11**, 52—64).—Transparent glasses were obtained in which soda of the parent glass $6SiO_2,1\cdot7Na_2O,0\cdot3CaO$ was molecularly replaced by zirconia in amounts up to 8% of the glass. A similar percentage of zirconia was also introduced into soda–magnesia–silica glasses. Series of glasses in which soda of the glass $6SiO_2$, $2Na_2O$ was successively replaced by 0·5 to 4 mols. of zirconia melted at temperatures from 1400° to 1450° and worked at 1150—1350°. High proportions of the oxide gave viscous glasses with a high rate of setting, and with increase of zirconia both annealing temperature and density increased. Similar results were obtained when silica on the parent glass was replaced by zirconia, and the glass $5SiO_2,ZrO_2,2Na_2O$ melted at 1500°, worked at 1350°, was very viscous, and set rapidly, The cubical thermal expansion constant for zirconia is $6\cdot9\times10^{-8}$, and the oxide is exceptionally valuable for producing resistance towards water, hydrochloric acid, and alkaline reagents.

A. COUSEN.

Function of arsenic in potash–lead oxide–silica glasses. S. ENGLISH, E. M. FIRTH, and W. E. S. TURNER (J. Soc. Glass Tech., 1927, **11**, 65—76).—To a normal crystal glass batch containing potassium nitrate, arsenious oxide was added in amounts up to 50 pts. per 1000 of sand, three types of batch being used with moisture contents (1) below 1%, (2) 1—2%, (3) 4%. Generally, 80—90% of the arsenic was retained, of which about 80% was in quinquevalent form, and these values were independent of moisture content. Progressive addition increased the rate of melting, but a maximum fining rate was reached with 20—30 pts. per 1000 of sand. With 2—5 pts. of arsenious oxide per 1000 of sand the most favourable melting and fining results occurred with 1—2% of moisture, with 10 pts. or more per 1000 variation of moisture did not affect melting and fining rates. Glasses from batches with 4% of moisture were the most viscous. The results with regard to retention of arsenious oxide applied also with lead crystal–phosphate opal glasses.

A. COUSEN.

Insulation of ceramic kilns. S. E. JOSI and A. E. HUBBARD (Trans. Ceram. Soc., 1926, **25**, 185—199).—The principles of heat insulation are outlined briefly, and the advantages of insulating kiln walls and crowns are pointed out. In tunnel kilns, insulating powder 9—12 in. thick is used over the crown, and a layer of insulating bricks 4—6 in. thick is built into the walls. The method of insulating periodic kilns is described and illustrated.

F. SALT.

Application of powdered coal as a tunnel-kiln fuel firing hard-fired common brick. F. M. HARTFORD (J. Amer. Ceram. Soc., 1926, **9**, 684—689).—Slack coal, ground in a ball-mill to pass 30-mesh, is passed with air to a distributing fan, so constructed that a mixture of powdered coal with about 30% of the air required for complete combustion is drawn over and distributed in equal parts to 10 burners. The stream of air and coal enters the rear of the burner, and is met by a horizontal stream of air for complete combustion.

F. SALT.

Specifications for lining and checker brick for water-gas manufacture. E. J. BRADY (J. Amer. Ceram. Soc., 1926, **9**, 667—678).—Tentative specifications for the purchase of refractories for the water-gas industry are presented, together with details of the process and equipment used in the industry, and tests prescribed in the specifications.

F. SALT.

Refractory materials of South Wales. W. R. D. JONES (Proc. S. Wales Inst. Eng., 1927, **43**, 115—140).—The occurrence, preparation, properties, and uses of silica, fireclay, and dolomite refractories of South Wales are discussed. The silica material is obtained from the basal grit of the millstone grit series ; the average silica content is about 95%, with 0·2—1·5% of alumina. The fireclays are obtained from the Lower Coal Series and the Pennant Series ; the silica content is 60—68%, and the alumina 20—25%.

F. SALT.

Spalling of bricks. F. W. PRESTON (J. Amer. Ceram. Soc., 1926, **9**, 654—658).—Norton's theory of spalling (B., 1925, 17) is criticised. The initial fissure in a typical case of spalling enters the brick normally to the isothermals, at right-angles to the tension, and makes its exit tangentially at other edges. Further, the isothermal planes, which are parallel to the face of the solid, are lines of principal stress, and therefore of zero shear. Once formed (at right-angles to the isothermals), the fissure advances in a direction at right angles to the lines of principal tension ; *i.e.*, the direction is related simply to the stress distribution at a given moment, but not to the temperature distribution. Diffusivity has no effect on the spalling tendency, but, on the other hand, the time elapsing between quenching and spalling is a function of the diffusivity.

F. SALT.

Effect of grain-size of fluxes and of non-plastic materials on the cone melting point of clays. H. SALMANG (Ber. Deut. Keram. Ges., 1926, **7**, 100—109).—Refractory tests were carried out on mixtures of kaolin and quartz ; kaolin and felspar ; kaolin, quartz, and felspar ; and kaolin and potash mica. The quartz, felspar, and mica were ground, and elutriated by decantation in glass cylinders into five fractions, varying in average grain-size from $2\cdot3\mu$ to 150μ. Two series of mixtures were prepared by adding these materials to kaolin to the extent of 25% and 50%. The tests were carried out at two different rates of heating, viz., 15° and 5° per min. The effect of reducing the grain-size of the fluxes and quartz on the m.p. was most marked at the rapid rate of heating ; in every case the slower heating tended to minimise this effect. In mixtures containing 50% of kaolin, 25% of felspar, and 25% of quartz, the m.p. was reduced from cone 31 to cone 27 by reducing the grain-size from 400μ to $2\cdot3\mu$.

F. SALT.

Effect of zinc oxide on the colouring properties of chromium, cobalt, and iron oxides. D. S. PILLAI (Trans. Ceram. Soc., 1926, **25**, 209—219).—Experiments

were carried out with three sets of triangular mixtures: cobalt, chromium, zinc; cobalt, iron, zinc; and chromium, iron, zinc. In this way 46 colour mixtures were obtained. The effect of various glazes on these mixtures was studied. Satisfactory colours were obtained from nearly all the mixtures, and they were suitable as under-glaze, on-glaze, and in-glaze colours, and as stains. Zinc oxide improved the cobalt blues, and formed a good range of browns, chocolate, and brownish-yellows with iron oxide. Chrome green is gradually destroyed by zinc owing to formation of zinc chromate. The green tints are improved, and the browns maintained with a magnesia glaze. F. Salt.

Use of opacifying media containing antimony in the enamel industry. H. Haupt and G. Popp (Z. angew. Chem., 1927, 40, 218—221).—Two "quinquevalent antimony compounds," sold under the trade names of "Leukonin" and "Timonox," were examined in order to determine whether, during the process of manufacturing cooking utensils, the antimony was reduced to the toxic tervalent form, and whether this was soluble in boiling liquids containing organic acids. Solubility in a boiling 3% solution of tartaric acid was determined on the two compounds separately in powder form, and on frits made from them and melted at 1400°. Enamelled ware was also prepared by adding the compounds (*a*) to the frit, and (*b*) to the mill mixture, and tested in 1% tartaric acid, 4% acetic acid, 1% potash solution, and water. Domestic conditions were also simulated by the use of boiling sauerkraut and apple sauce. Leukonin was practically free from tervalent antimony, whereas Timonox contained this form only. The above methods of preparing Leukonin enamels had little effect on the solubility; a small proportion of the quinquevalent antimony is always reduced to the tervalent form, traces of which go into solution with tartaric acid. The quantity, however, is so minute as to be hygienically harmless. On the other hand, Timonox is quite unsuitable for the manufacture of domestic ware. F. Salt.

Resistance of enamel to attack by mineral acids. A. Kerstan (Sprechsaal, 1926, 59, 708—709; Chem. Zentr., 1927, I, 342).—Enamel is attacked most readily by hydrochloric acid and nitric acid when the acids are of 25% and 35% strength respectively. Hydrochloric acid attacks the borates and part of the alkalis; nitric acid dissolves principally sodium oxide, potash enamels being far more resistant to its action. L. A. Coles.

Glass wool as insulator for refrigeration purposes. H. C. Bates (J. Amer. Ceram. Soc., 1926, 9, 690—692).—The insulating properties of glass wool of various densities, powdered cork, magnesia–asbestos composition, slag wool, Sil-o-Cel powder, cotton waste, and sheep's wool were determined by observing the rate at which ice melted in a vessel insulated with these materials. The change in volume of a mixture of ice and water was observed by means of a graduated tube leading from the vessel. The most effective packing for glass wool was that corresponding to a density of 0·065 g./c.c. The various conductivity values are tabulated, showing the high value of dry glass wool as a heat insulator. F. Salt.

Determination of silica in refractories. Stadeler.—See X.

Patents.

Production of refractory material. C. Deguide (F.P. 612,094, 19.6.25).—Mixtures of barium silicate containing from 1 mol. or more of barium oxide per 1 mol. of silica, with water-glass, alumina, silica, tar, and water, are moulded and calcined, yielding bricks suitable for lining furnaces and cement kilns. L. A. Coles.

Clay bodies or material for making tiles, slabs, sanitary and other ware. W. Gourdjian and G. L. Jones (E.P. 268,204, 30.7.26).—Gum arabic is incorporated with the clay to produce when fired a hard ceramic article. B. W. Clarke.

Manufacture of blue glazed ceramic articles with the appearance of old Egyptian ware. M. Pick (G.P. 436,182, 7.6.24).—A glaze consisting of water-soluble alkali salts with the addition of copper oxide is burnt at a low temperature on a low-burnt body rich in silica. The subsequent glazing is carried out at a temperature sufficiently low to prevent the complete fusion of the silica in the glaze. B. W. Clarke.

Metallising glass surfaces. Soc. Nouvelle de Métallisation (F.P. 612,401, 27.6.25).—The metal is sprayed by Schoop's process on to the glass surface coated with a layer of gelatin or similar material mixed with glycerin, at such a temperature that it is of a suitable consistency, and the layer is hardened by treatment with formaldehyde. L. A. Coles.

Glass furnace. United Glass Bottle Manufrs., Ltd., and T. C. Moorshead (E.P. 268,432, 31.12.25).

Manufacture of sheet glass. A. E. White. From Pittsburgh Plate Glass Co. (E.P. 269,300, 9.3.26).

Rouge (U.S.P. 1,618,086).—See VII.

IX.—BUILDING MATERIALS.

Report of the Stone Preservation Committee (Dept. Sci. Ind. Res., March, 1927. 33 pp.).—A detailed examination of the causes of decay in stone is being made before attempting to devise or recommend methods of preservation. The microstructure of building stones, as revealed by an examination of sections mounted in a specially developed synthetic resin, is apparently of primary importance, and the distribution of pores in the stone, *i.e.*, of the surface open to attack, is probably a preponderating factor in determining the rate of attack. Lack of information on this point in previous researches renders correlation of results of other workers very uncertain, and probably accounts for the widely differing results obtained in practice with various methods of preservation, *e.g.*, limewashing. Experiments are being made on test pillars treated with various preservatives which appear to offer the highest initial resistance to attack. A preliminary bacterial investigation has led to the isolation of a special strain apparently only found in stonework. B. W. Clarke.

Action of potash end-liquors and mother-liquors on cements. Calame (Kali, 1926, **20**, 328—336; Chem. Zentr., 1927, I, 342).—Cement with a low lime content, such as blast-furnace cement, is most resistant to attack by the liquors, and its properties in this respect are improved if Portland cement having a high resistance to attack by the liquors is used in its manufacture Blast-furnace cement containing not too much clinker, mixed with about 10% of trass, is recommended.

L. A. Coles.

Hardening of roads containing silicates. R. Feret (Compt. rend., 1927, **184**, 935—937).—Stony or calcareous materials are rendered hard by a solution of sodium silicate (SiO_2 24·9%; Na_2O 7·3%) owing to the separation of gelatinous silica which subsequently coagulates, and binds the particles of stone into a resistant and insoluble mass. This occurs by means of adsorption, and since it is favoured by drying, dialysis, and the action of carbon dioxide, the different effects obtained under various working conditions may be explained. J. Grant.

Use of p_H colorimetric reagents for the recognition of green or dry wood. R. Legendre (Compt. rend., 1927, **184**, 949).—As indicated by the use of methyl red, the p_H values for macerated wood vary between 4·4 and 6·0, according to the age of the wood.

B. W. Anderson.

Patents.

Composition for building walls, floors, and structures. I. F. Shellard (E.P. 268,633, 27.8.26).—Portland cement (1 pt.) is mixed with 5 pts. of an aggregate consisting of 1 pt. of clean stone chippings and 4 pts. of clayey stone dust, arranged so that the aggregate contains just over 5% of clay. B. W. Clarke.

Production of cast articles from volcanic stones or like materials or mixtures thereof. C. Krüger (E.P. 262,413, 22.11.26. Conv., 2.12.25).—Volcanic stone, blast-furnace slag, or the like is heated so that only the non-crystalline matrix and the easily fusible crystals are fused, the difficultly fusible crystals remaining to serve as crystallisation centres on cooling, with the addition of other minerals not readily fusible to act as further centres if required. B. W. Clarke.

Acid-resisting cement. Schmelzbasalt-A.-G., and C. Trenzen (G.P. 435,913, 7.7.25).—Quartz, quartzite, or other acid-resisting material is mixed with a colloidal solution of water-glass (*d* 1·308—1·332) and barium hydroxide, with or without the addition of assistant or protective colloids. D. Woodroffe.

Manufacture of material similar to compressed asphalt. H. H. Schroder (E.P. 260,621, 30.10.26. Conv., 2.11.25).—Limestone marl having a sp. gr. about half that of compact rock limestone is intimately mixed in a granulated form, at a temperature above normal, with sufficient bitumen to obtain an incompact mass, which is moulded in the cold state and under moderate pressure to form an asphalt-like substance suitable for use as a road surface and the like. B. W. Clarke.

Concrete block composition. M. E. von Mach, jun. (U.S.P. 1,623,985, 12.4.27. Appl., 13.4.22).—The residue of burned fuel is treated with a mineral acid to remove sulphides, washed to remove soluble matter, and mixed with cement, finely-divided wood, lime, and plaster of Paris. B. W. Clarke.

Bituminous emulsions [for road-making]. F. Levy (E.P. 268,411, 3.12.25).—Bitumen is heated at 85—107° and agitated with a hot dilute aqueous solution of sodium silicate, forming an emulsion without the aid of fatty or saponifiable matter.

B. W. Clarke.

Apparatus for drying and seasoning timber. A. M. Kobiolke (U.S.P. 1,625,548, 19.4.27. Appl., 25.7.23. Conv., 25.7.22).—Steam from a boiler is distributed throughout a hermetically sealed kiln which is connected to a vacuum pump, and is provided with a condenser to cause condensation inside the kiln.

B. W. Clarke.

Facing or preserving walls and other surfaces. J. W. Dougal (E.P. 268,016, 21.12.25).

Cement and lime burning. G. E. Heyl (U.S.P. 1,625,853, 26.4.27. Appl., 4.3.26. Conv., 5.3.26).—See E.P. 252,780; B., 1926, 668.

[Corrugated asbestos-cement] roofing and covering materials for buildings. C. C. Field (E.P. 268,499, 10.2.26).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Gaseous reduction of iron ores. H. Kamura (Trans. Amer. Electrochem. Soc., 1927, **51**, 413—422).—Iron ores are rapidly reduced by carbon monoxide at about 900°, giving a very pure sponge iron relatively free from silicon, manganese, and phosphorus. The reduced mass may be pressed into briquettes for melting in the electric or open-hearth furnace, giving a pure malleable iron or steel of any desired composition, and a slag containing all the impurities. Theoretical calculations substantiated by experiments show that the fuel consumption in the process would be about 0·3 ton of coke and 0·5 ton of coal per ton of iron produced, compared with 1 ton of coke per ton of iron in the usual blast-furnace process. A. R. Powell.

Composition of materials from various elevations in an iron blast-furnace. S. P. Kinney (U.S. Bur. Mines, Tech. Papers, 1926, No. 397. 22 pp.).—In a blast-furnace working southern red and brown ores and pyrite sinter, 80—85% of the iron was reduced by the time it had reached the base of the shaft, 19¼ ft. above the tuyère level. The sponge iron at that point contained considerable impurities, *i.e.*, more than one third of the carbon and manganese and one half of the phosphorus content. Most of the silicon entered the metal in the bosh of the furnace, and was increased rapidly in the tuyère zone by the oxidation of part of the manganese, the content of which was reduced from 1·1% to 0·53% in the cast. More (0·07%) than the tolerant amount of sulphur had been absorbed at the base of the shaft, increasing to twice the tolerant amount (0·05%) at a point 27 in. above the tuyère level. Desulphurisation

occurred between this point and the tuyère. Preferential absorption of sulphur by lime in the upper part of the furnace was demonstrated. C. A. KING.

Occurrence, distribution, and significance of alkali cyanides in the iron blast-furnace. S. P. KINNEY and E. W. GUERNSEY (U.S. Bur. Mines, Tech. Papers, 1926, No. 390. 37 pp.).—The cyanide content of measured quantities of blast-furnace gas drawn from different planes of a blast-furnace by means of a water-cooled sampling tube was determined. At the tuyère level and also on a plane 27 in. above, the concentration of cyanides per 1000 cub. ft. of gas ranges from nothing at the wall to 3—4 oz. at the centre, about 3 oz. at the base of the shaft, and 10—20 oz. at the slag notch, although wide variations due to condensation occurred. Considering the probable effect of withdrawing a portion of the gas for cyanide recovery there is good reason to believe that the rate of formation of cyanide is determined largely by the rate at which alkali can be displaced from silicates, and that the cyanide content of the gases where cyanide is being formed is much below the concentration for equilibrium with the liquid phase in the charge. It seems unlikely that the furnace operation would be affected adversely by the withdrawal of a small part of the gas, and it is estimated that the daily recovery of crude cyanide would be 1040 lb. if one tenth of the gas at the base of the shaft was withdrawn, a recovery which would be commercially significant.

C. A. KING.

Influence of rust-film thickness upon the rate of corrosion of steels. E. L. CHAPPELL (Ind. Eng. Chem., 1927, **19**, 464—467).—The rate of corrosion of a steel in the atmosphere depends on its chemical constitution, and the thin film of rust formed has no protective effect. A number of such films on steels of varying composition after 6 years' exposure were all found to be 0·0005 cm. thick. Tests with an artificial covering of filter paper of varying thickness showed that, with films over 0·02 cm. thick, the corrosion is inversely proportional to the thickness and independent of the chemical composition. Such films only occur naturally in steel permanently under water. For such conditions, unless the rust film is removed, which is inadvisable, the chemical constitution of the steel is of no importance.

C. IRWIN.

Determination of sulphur in steel. W. HERWIG (Chem.-Ztg., 1927, **51**, 275).—A battery of vertical glass tubes has delivery tubes leading into potassium hydroxide solution. Hydrogen sulphide evolved on dissolving the steel samples in a hydrochloric–sulphuric acid mixture in the battery is trapped in the potassium hydroxide, and is then determined by acidification and titration with iodine. The temperature of the dissolution tubes is kept at 110—120° by immersion in an oil bath. The time of operation is from 10—15 min. L. M. CLARK.

Utilisation of manganiferous iron ores. T. L. JOSEPH, P. H. ROYSTER, and S. P. KINNEY (U.S. Bur. Mines, Tech. Papers, 1926, No. 393. 28 pp.).—Manganiferous iron ores from the Cuyuna district of Minnesota, which have a high content of phosphorus and alumina, and are low in silica, have been reduced in an experimental blast-furnace 31 ft. high and 6½ ft. in diameter at the bosh. No difficulty was experienced in operating with a burden of 100% of brown ore, though careful adjustment of the charge was necessary on account of the high ratio of alumina to silica. The metal contained 11·68—14·4% and the slag 4·8—12% Mn. There was considerable variation in gas composition across the furnace, and the experiment is not considered to be completed. The utilisation of pig iron such as was produced in the experimental blast-furnace depends on the elimination of phosphorus, which averages 0·6%, and it is suggested that this might be accomplished by either a converter process or a duplex process using a converter and an open-hearth. C. A. KING.

Influence of molybdenum on medium-carbon steels containing nickel and chromium. J. A. JONES (Res. Dept., Woolwich, R.D. Rept., 1926, No. 67. 59 pp.).—The examination of a number of nickel, chromium, and nickel–chromium steels containing molybdenum, showed that increase in molybdenum content slightly raised Ac1 and lowered Ar1, the quantitative effect of the latter influence varying considerably in different steels, though the initial temperature is without effect on the position of the Ar1 point. When the rate of cooling is slow molybdenum effects more perfect hardening than nickel or chromium, and mass effect is less marked, thereby giving greater uniformity of properties throughout the walls of large forgings after treatment. Although the changes in the properties of steel due to the presence of molybdenum are progressive with increase of molybdenum, the most marked effect is produced in nickel–chromium steels by the addition of not more than 0·5% Mo. Generally, the presence of molybdenum improves the values of mechanical tests and reduces the susceptibility of steel to temper brittleness. The best combination of properties is provided by nickel–chromium–molybdenum steels, closely approached and in certain particulars exceeded by nickel–molybdenum and chromium–molybdenum steels. An appendix contains the detailed results of a large number of experiments.

C. A. KING.

Decomposition of austenite during quenching. R. L. DOWDELL and O. E. HARDER (Trans. Amer. Soc. Steel Treat., 1927, **11**, 217—232).—Tests were made on 10 high-carbon or alloy steels which were quenched from high temperatures to retain the maximum amounts of austenite. The results show that quenching in oil produces more austenite than the more drastic quenching in water, which, owing to greater temperature differences between the centre and periphery, causes higher tensional stresses at the edges and assists the formation of martensite. A compression stress in the core or the absence of tension favours the retention of austenite. If the core is a region of tensional stresses the structure is martensitic throughout. On quenching from a higher temperature the austenite grains are larger and more stable, the stability being further increased by the larger amount of carbon taken into solution by the austenite.

T. H. BURNHAM.

Rapid analysis of brass. S. VON BOGDANDY and M. POLANYI (Z. Metallk., 1927, **19**, 164—165).—On heating brass at 1190° in a vacuum the zinc and lead are completely distilled out of the metal, leaving a residue of

copper with traces of iron. The operation is carried out in a silica tube in the lower end of which is placed a small silica test-tube containing 0·15—0·2 g. of the brass. The upper part of the outer tube is connected to the vacuum pump and water-cooled, whilst the lower part is placed in a platinum-wound, electric resistance furnace previously heated at 1190°. After 5—6 min. the tube is removed from the furnace, cooled in water, and the copper button weighed. By repeating the process on the sublimate, but using a temperature of 600°, the zinc alone distils, leaving a residue of lead. The results for copper in a plain brass are within 0·1% of the theoretical.

A. R. POWELL.

Controllable variables in the submerged corrosion of metals. O. B. J. FRASER, D. E. ACKERMAN, and J. W. SANDS (Ind. Eng. Chem., 1927, **19**, 332—338).—The corrosion rate of monel metal in 5% sulphuric acid solution varies directly with oxygen concentration, reaching a maximum when the solution is saturated in oxygen. Increase in the rate of aeration causes a rapid increase in the corrosion up to a point, after which further increase has little effect. With increasing sulphuric acid concentration a maximum corrosion rate is reached in 5% of acid, the rate then decreasing until, with 80% of acid, an abrupt and rapid increase indicates a change in the corrosion reaction. The rate of corrosion increases with the velocity of relative movement between the test piece and the solution, but the rate of change gradually diminishes, and tends to become constant when the velocity is 200 ft./min. With increasing temperature, the rate of corrosion increases logarithmically to a maximum at 70°, after which the rate diminishes owing to the rapid decrease in the solubility of oxygen at the higher temperatures. The finish of the test-piece surface has little effect on the corrosion rate. L. M. CLARK.

Heavy liquids for mineralogical analyses. J. D. SULLIVAN (U.S. Bur. Mines, Tech. Papers, 1926, No. 381. 26 pp.).—A review of the sp. gr. and uses of heavy liquids and mixtures for use in determining the density of, and separating, minerals. Bromoform–carbon tetrachloride or acetylene tetrabromide–carbon tetrachloride, *d* 1·58—2·89; stannic bromide–carbon tetrachloride, *d* 1·58—3·35; tin tetrabromide–antimony tribromide, *d* 3·11—3·64; thallous silver nitrate solution, *d* 1—4·6; thallous formate solution, *d* 2·86—4·94; and mercuric chloride–mercuric iodide–antimony trichloride, *d* up to 5·2, are recommended. For routine work the Mississippi Valley Station of the Bureau of Mines have simplified practice to two heavy liquids only. Acetylene tetrabromide is recommended for all low-gravity work, and thallous formate–malonate, which is miscible in water, for work on minerals of high density (*d* 4·3). As a less expensive reagent, thallous formate may be used up to *d* 3·5. C. A. KING.

Recovery of molybdite from the ore. H. A. DOERNER (U.S. Bur. Mines, Tech. Papers, 1926, No. 399. 13 pp.).—All the known deposits of molybdenum contain considerable quantities of molybdite occurring as an oxidation product of molybdenite which is recovered readily by flotation. A number of reagents were tested under a great variety of conditions, and in no test was there any indication that molybdite could be floated. Gravity methods of separation are obviously useless because of the low sp. gr. and softness of molybdite, nor did the dielectric method (cf. Hatfield; B., 1924, 261) prove promising. Two leaching methods showed the possibility of larger scale operations in which (A) the ore is extracted with a hot solution of sodium carbonate, acidified with sulphuric acid, a small quantity of sodium acetate being added, and digested with lead sulphate; molybdenum is completely precipitated as lead molybdate which can be converted to molybdic acid, or (B) the ore is digested with warm dilute sulphuric acid, and the solution neutralised with scrap iron, molybdenum being precipitated as a hydrate of the lower oxide, MoO_2. The separation of molybdenum from all other constituents of the ore is practically complete by treating the roasted ore at 450° with chlorine.

C. A. KING.

Determination of phosphorus in phosphorus alloys. B. SALKIN (Ind. Eng. Chem., 1927, **19**, 416—417).—The alloy is dissolved in dilute aqua regia solution, and any phosphine escaping is absorbed in a bromine-concentrated nitric acid solution. The two solutions containing phosphorus are combined, and the usual procedure for determination of phosphorus in such solutions is followed. L. M. CLARK.

Sources of error in the determination of phosphoric acid by the molybdate–magnesia method. II. J. M. MCCANDLESS and J. I. BURTON (Ind. Eng. Chem., 1927, **19**, 406—409; cf. B., 1925, 80).—Magnesium ammonium phosphate precipitated by addition of magnesia mixture to a hydrochloric acid, ammoniacal, or neutral solution of ammonium phosphomolybdate always contains molybdenum, the purity being greatest in the precipitate from neutral solution. The more concentrated the solution, the greater will be the amount of molybdenum carried down. Ignition at 1000° volatilises most of the molybdenum. When the residue is grey or black after ignition it still contains molybdenum, which can only be volatilised with a loss of phosphoric acid. It is recommended that the precipitate should be filtered on an asbestos mat in a platinum Gooch crucible, and heated to constant weight in a blast-flame. Molybdenum in magnesium pyrophosphate is determined colorimetrically as sulphide by comparison with standards. Microcosmic salt is suggested as a phosphate standard. L. M. CLARK.

Determination of silicon and silica in aluminium. PRETTNER (Chem.-Ztg., 1927, **51**, 261).—Methods for the determination of silicon combined and free in aluminium alloys, which depend on removal of silica from a mixture of silicon and silica by treatment with hydrofluoric acid, are critisised, since silicon deposited from solution when an alloy is chilled readily oxidises on ignition. The mixture of silicon and silica is preferably dried at 200°.

L. M. CLARK.

Determination of silica in ores, slags, fluxes, and refractory materials containing fluorine. A. STADELER (Stahl u. Eisen, 1927, 47, 662—664).—Comparative tests on five methods of determining silica in materials containing fluorine showed that fusion with alkali carbonate followed by precipitation of the silica with ammonium carbonate and ammoniacal zinc oxide

solution gives the most accurate results. Good results are obtained by fusion with alkali carbonate followed by evaporation with hydrofluoric acid only when the material contains less than 1% of fluorine. For the determination of silica in fluorspar the powdered mineral is extracted with acetic acid, and the residue calcined at 800°, weighed, evaporated with hydrofluoric acid, again heated at 800°, and weighed again; the difference is silica. A. R. Powell.

Selenium sulphide. A detector for mercury vapour. B. W. Nordlander (Ind. Eng. Chem., 1927, **19**, 518—521).—A solution of aluminium chloride containing 100 mg. of aluminium per litre is saturated with hydrogen sulphide. At the same time selenious acid is run in. The yellow precipitate is collected, washed, and dried, and is applied to smooth, dense, but not highly glazed paper with cotton wool. This paper is darkened by mercury vapour, the shade varying with the concentration, time of exposure, and temperature. At a velocity of 1 m./sec. and a temperature of 70°, 1 pt. of mercury in 4 million pts. of air give a colour change in 4 min. A machine devised to measure similar concentrations by the time required to match a shade is described. The test is specific for mercury liquid or vapour, and is not given by mercury ions in solution. C. Irwin.

Determination of mercury vapour. B. W. Nordlander (Ind. Eng. Chem., 1927, **19**, 522—524).—The attempt to remove mercury vapour from air by agitation with a spray of dilute aqua regia leaves the content of mercury in the air (as shown by selenium sulphide; cf. preceding abstract) almost unchanged. A similar result was found when the air was passed through a weighed glass tube filled with gold leaf and glass wool and the amalgamated mercury weighed. In both cases the proportion of mercury removed was about 0·2% of that present, and the methods lead to totally erroneous results. C. Irwin.

Patents.

Heat-treatment of alloy steels. P. R. Kuehnrich (E.P. 267,687, 19.2.26).—A process for hardening and imparting stainless qualities to steel containing 10—20% Cr and 1—1·7% C, with or without other metals in addition to iron, consists in heating it out of contact with air and preferably in a salt bath at 1150°, cooling in air, reheating at 650—700°, air-cooling, reheating in a salt bath at 1020°, and quenching in paraffin, oil, or water. C. A. King.

[Rustless cast iron] alloy. P. D. Merica and J. S. Vanick, Assrs. to Internat. Nickel Co. (U.S.P. 1,626,248, 26.4.27. Appl., 26.9.25).—Cast iron containing carbon within the usual ranges is rendered corrosion-resistant by the addition of 5—35% Ni and 2—15% Cr, the percentage of nickel exceeding that of the chromium. F. G. Crosse.

Steel of high-temperature stability. W. R. Shimer, Assr. to Bethlehem Steel Co. (U.S.P. 1,624,075, 12.4.27. Appl., 29.11.24).—A low-carbon, tough steel contains 0·4—0·6% Cr, 0·5—0·8% Mn, and also 1—1·5% W to preserve the strength of the steel at high temperatures. C. A. King.

Copper alloys. M. G. Corson (E.P. 268,200, 21.7.26).—Copper alloys which do not anneal at relatively high temperatures contain 0·3—2% Cr or 1—6% Co. The alloy is quenched at 900—975°, and is then subjected to mechanical working. C. A. King.

Copper alloys. Metallbank u. Metallurgische Ges. A.-G. (E.P. 268,225, 21.9.26. Conv., 15.4.26).—An alloy suitable for bearings contains 78·5—93·8% Cu, 5—9·5% Al, 1—6% Mn, 0·1—3% Fe, and 0·1—3% Ni. C. A. King.

[Aluminium] alloy. A. Pacz, Assr. to Aluminum Co. of America (U.S.P. 1,595,058, 3.8.26. Appl., 19.12.24).—An aluminium base alloy contains 3—15% Si, 1·0—1·5% Cu, and 0·5% Mn. F. Salt.

Treatment of zinc ores and other zinciferous materials. W. Job (E.P. 268,201, 22.7.26).—Zinc ores, or zinciferous materials containing lead, copper, or noble metals are smelted in a blast furnace to volatilise lead and part of the zinc. Copper and the noble metals are recovered in the form of a matte, and the zinc-bearing slag is smelted again. In a shaft furnace suitable for this process the hearth is constricted so that the distance between opposite tuyères may be less than 1 m. The shaft above the tuyère zone is bulged out so that the gases pass up the centre of the furnace, and smelting of the charge near the side walls is largely prevented. Additional air is supplied to the upper part of the furnace (cf. U.S.P. 1,526,910; B., 1925, 323). C. A. King.

Extraction of lead and zinc from ores and metallurgical products. F. Krupp Grusonwerk A.-G. (E.P. 256,601, 24.7.26. Conv., 8.8.25).—Ore containing lead and zinc is heated under oxidising conditions in a rotary furnace to volatilise lead oxide, and the residue, mixed with reducing agents, is further heated to volatilise zinc which is collected separately (cf. E.P. 24,009 of 1910; B., 1911, 1320). C. A. King.

Tin-plate and sheet-mill furnace. R. Thomas & Co., Ltd., and T. F. Davies (E.P. 267,592 16.12.25).—The heating chamber of a sheet-mill furnace is protected from the direct action of flames from the combustion chamber by what is essentially a false crown extending from the bridge at least two thirds of the length of the furnace. Inspection apertures are provided through the arch and bridge extension, both of which incline downwardly toward the exit port. Subsidiary ports may be provided at each corner of the furnace bottom connecting with the main flue which underlies the bed of the furnace. C. A. King.

Electrolytic deposition of cadmium. E. C. R. Marks. From Grasselli Chemical Co. (E.P. 265,292, 4.11.25).—Cadmium may be deposited from a solution containing a cadmium compound (*e.g.*, the oxide, hydroxide, cyanide, or sulphate), a cyanide of cadmium, ammonium, or other metal, an ammonium compound such as ammonia solution, the cyanide, or sulphate, and an alkaline reagent such as ammonia solution or the hydroxide of an alkali metal. The ammonium salt tends to prevent the deposition of other metals, such as zinc, along with the cadmium. The bath may also contain a colloidal substance such as starch or gulac, which helps to improve the quality of the cadmium

plate. The electrolysis is conducted under carefully regulated conditions of concentration of electrolyte, temperature, voltage, and current density. The surface area of the cadmium anodes should be 2—3 times that of the cathodes. The process may be adapted to the determination of cadmium in the presence of zinc.

M. E. NOTTAGE.

Cleaning metal. S. R. MASON, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,625,484, 19.4.27. Appl., 7.6.23).—Metal is subjected first to an alternating current in an electrolyte of ferric chloride and hydrochloric acid, and then, after removing the scale and rust, as an anode to direct current in a solution of sulphuric acid. H. ROYAL-DAWSON.

Apparatus for determining the percentage of carbon in iron or steel. C. J. G. MALMBERG and J. G. HOLMSTRÖM (U.S.P. 1,626,005, 26.4.27. Appl., 26.6.24). —See E.P. 184,215; B., 1922, 763A.

Treatment of steel ingots. J. V. ELLIS, and UNITED STEEL COMPANIES, LTD. (E.P. 268,123, 18.3.26).

Hearths of blast and like furnaces. E. BARTEN (E.P. 267,468, 30.11.26. Conv., 9.3.26).

XI.—ELECTROTECHNICS.

PATENTS.

Electric furnace. F. T. COPE, Assr. to ELECTRIC FURNACE Co. (U.S.P. 1,624,217, 12.4.27. Appl., 9.11.25). —A hearth comprising a number of plates having lugs at opposite edges for engagement under the edges of adjoining plates is spaced above a refractory base, and a resistor is mounted between the hearth and base.

J. S. G. THOMAS.

Affixing [metallic oxide] coatings on incandescence lamp filaments. H. K. RICHARDSON, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,624,071, 12.4.27. Appl., 28.7.21).—A high potential is established between the filament and another electrode connected thereto, in a rarified atmosphere in the presence of the powdered metallic oxide, so as to effect bodily transfer of the powdered particles to the filament without dissociation.

J. S. G. THOMAS.

Thermionic electrode. BRITISH THOMSON–HOUSTON Co., LTD., Assees. of G. M. J. MACKAY (E.P. 267,142, 4.3.27. Conv., 6.3.26).—In a thermionic electrode of the type described in E.P. 208,728—9 (B., 1925, 391), a metal container, *e.g.*, of nickel, encloses a core of film-forming material, *e.g.*, cæsium, rubidium, barium, or calcium, of materially lower electron affinity, which is able to diffuse through the wall of the container at high temperatures. Alternatively, the core may consist of a mixture of a compound of one of the said metals, *e.g.*, cæsium chloride, and a reducing agent, *e.g.*, powdered magnesium or calcium, so that at the operating temperature of the electrode the desired metal is produced without the formation of deleterious products.

J. S. G. THOMAS.

High-pressure system for electrolytic processes, more especially for the production of hydrogen and oxygen. J. E. NOEGGERATH (E.P. 268,426, 30.12.25).—An electrolyser adapted for high-pressure electrolytic systems has a cross-section so reduced in area as to be only sufficient to accommodate the gas bubbles passing through it when operating at high pressure. The vessel consists of a number of individual cells connected in series, the distance between electrodes of different polarity being greater at the bottom than at the top, whilst, in order to obtain smaller electrolyte-filled spaces, parts of the vessel are insulated against the electrolyte, so that the walls of the vessel can be used for conducting current. Partitions separating electrodes are made of asbestos cord, and yield to the variations of volume due to the different pressures of the gases. W. G. CAREY.

Galvanic battery. C. DE MIERES (E.P. 268,047, 28.12.25).—A secondary battery comprises a positive electrode of graphite, artificial carbon, or other electrical conductor not attacked by chlorine or bromine, covered with spongy silver, a narrower and shorter negative electrode of zinc, and an electrolyte consisting of an aqueous solution of zinc chloride mixed with one or more other halogen salts of zinc, *e.g.*, zinc bromide. The voltage of the cell is 2·2. J. S. G. THOMAS.

Impregnating porous substances or fillers with artificial resins. BAKELITE GES.M.B.H. (F.P. 611,264, 15.2.26. Conv., 17.2.25).—The material is impregnated with a colloidal solution of artificial resin, *e.g.*, phenol–formaldehyde condensation products, and the resin precipitated by means of water. Thus, sawdust is mixed with a 50% solution of artificial resin, then treated with water, centrifuged, and dried. Similarly, paper pulp, fibrous material, asbestos, or mica may be treated with a colloidal suspension of artificial resins and soap solution. The product is suitable for making electric insulators etc. and for lining chemical plant. J. S. G. THOMAS.

Electric arc welding or coating with material. INTERNAT. GENERAL ELECTRIC Co., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (E.P. 262,453, 3.12.26. Conv., 3.12.25).

Production of electrodes for electric accumulators. P. HAGSPIHL (E.P. 265,918, 22.10.26. Conv., 13.2.26).

Manufacture of [plates for] accumulators or electrical storage batteries. S. J. WILLIAMS (E.P. 268,179, 28.5.26).

Gas analysis apparatus (E.P. 268,637).—See I.

Apparatus for determining impurities in water (E.P. 268,597).—See I.

XII.—FATS; OILS; WAXES.

Marine animal oils. I. Squalene and spinacene. E. ANDRÉ and H. CANAL (Ann. Chim., 1927, [x], 7, 69—112).—A detailed account of an investigation, a preliminary note of which has already appeared (A., 1925, i, 1373). The liver oil from *Cetorhinus maximus*, *Scymnus lichia*, and *Centrophorus granulosus* was separated by hydrolysis into fatty acids, alcohols, and non-hydrolysable matter, physical and other constants of each fraction from the various specimens being given. Conversion of the unsaturated hydrocarbons from the three species, after purification by fractional distillation under reduced

pressure, into hydrochlorides and hydrobromides, and fractional crystallisation of these from acetone yields, respectively, hydrochlorides, $C_{28}H_{46}$,6HCl, m.p. 107—108°, and $C_{30}H_{50}$,6HCl, m.p. 143—145°; $C_{30}H_{50}$,6HCl, m.p. 107—108°, and $C_{31}H_{52}$,6HCl, m.p. 143—145°; $C_{30}H_{50}$,6HCl, m.p. 106—107°, and $C_{31}H_{52}$,6HCl, m.p. 141—143°; *hydrobromides*, $C_{28}H_{46}$,6HBr, m.p. 106°, and $C_{30}H_{50}$,6HBr, m.p. 130—132°; $C_{28}H_{46}$,6HBr, m.p. 105°, and $C_{30}H_{50}$,6HBr, m.p. 130—132°; $C_{29}H_{48}$,6HBr, m.p. 105°, and $C_{30}H_{50}$,6HBr, m.p. 132—134°. As a result of a large number of analyses by different methods, it is found that the hydrohalide with the lower m.p. always contains a larger proportion of halogen, but traces of hydrogen halide are lost during the crystallisations, and that, moreover, the addition of hydrogen halide to the six ethylenic linkages is not complete, a carefully purified sample of squalene hydrochloride, the analysis of which corresponded with a C_{30} formula, losing sufficient halogen after several months to agree with a C_{31} formula. Re-treatment of this sample with hydrogen chloride in ether gave a specimen corresponding with C_{29}, and in acetone, with one corresponding with the formula $C_{28}H_{46}$,6HCl. These facts show that the constitution of the hydrocarbon cannot be determined by analysis of the hydrohalides alone (cf. Heilbron, Kamm, and Owens; A., 1926, 816, whose work is criticised on this ground), the authors holding the view that the formula $C_{28}H_{46}$ (or even $C_{27}H_{44}$ from its relation to cholesterol) for squalene is more probable than that of higher homologues. J. W. Baker.

Determination of the iodine value according to the German Pharmacopœia. VI. K. H. Bauer and P. Manicke (Pharm. Zentr., 1927, **68**, 241—250).—Comparative determinations of the iodine values of drying, semi-drying, and non-drying oils and fats by various methods are described. The Hanus method gave results lying within the limits given in the G.P. VI., and with 33—100% excess of halogen no notable differences were observed, except with cod-liver oil, which, with 33% excess, gave an iodine value of 160·9, and with 100% excess, 164·7. Reaction periods varying from 15 min. to 2 hrs. produced no marked variation in the results. The Kaufmann method gave values agreeing generally with those given by the Hanus method. Varying excesses of halogen had no influence on the iodine value except with linseed oil, which gave higher values with excess. The pyridine sulphate dibromide method of Rosenmund–Kuhnhenn gave rather lower values than the above two methods, even with large excess of halogen and longer reaction period. Winkler's method—the official G.P. VI. method—gave results with hard fats agreeing with those given by the Hanus and Kaufmann methods, but the oils gave generally higher values. The excess of halogen used varied from 15% to 200%, and a more satisfactory end-point was obtained by determining the excess of halogen by the addition of potassium iodide and titration with thiosulphate. The method gave results falling within the official limits, but it is doubtful if it is superior to either the Hanus or the Kaufmann method. E. H. Sharples.

Patents.

Preservation of oils. F. W. Nitardy, Assr. to E. R. Squibb & Sons (U.S.P. 1,624,358, 12.4.27. Appl., 21.1.24).—Castor oil, from which odour and taste have been removed, and containing a small proportion of ethyl alcohol, is claimed. S. S. Woolf.

Manufacture of soap [filaments]. A. H. Charlton, and T. B. Rowe & Co., Ltd. (E.P. 267,999, 24.11.25).—Liquid soap, preferably containing about 63% of fatty acids, with or without the addition of sodium carbonate, sodium silicate, etc., is forced through suitable orifices under a pressure of 15—60 lb./sq. in., and the solidified filaments so formed are subsequently dried. The liquid soap may be aerated before extrusion. S. S. Woolf.

Pattern printing using wax (Austr. P. 104,395).—See VI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

"Oxyns." IV. The autoxidation process. A. Eibner and H. Munzert (Chem. Umschau, 1927, **34**, 89—96, 101—108; cf. B., 1926, 888).—Previous work and existing theories on the processes involved in the oxidation of drying oils are comprehensively and critically recapitulated. In developing the concept of "ideal drying of an oil," the various factors influencing a drying oil film are discussed, and an expression is devised analysing the total increase in weight of a film into its components due to these separate influences. The gradual increase of acidity is shown to be due to the hydrophilic nature of the drying film. Evidence is adduced for the absence of true hydroxy-acids in "oxyn acids," but reduction, acetylation, benzoylation, etc. of these fail to yield crystallisable products that would lend themselves to further investigation. The oxyn acids are probably non-hydroxylic intermediates between peroxy-acids and true hydroxy-acids (contrary to the views of Auer), and various possibilities of tautomerism are discussed. Dried oil films and artificially oxidised oils, blown oils, Uviol oils, etc. ("liquid oxyns") yield dark-coloured soaps and acids on saponification and acidifying, whereas the soaps and acids obtained from fresh oils and stand oils are of lighter colour. True hydroxy-acids of the C_{18} group and their salts are colourless, crystalline, and water-soluble. Tung oil is the most suitable oil for experimental use owing to its freedom in the main from mixed glycerides, and its capabilities of yielding purifiable products, and its so-called anomalous behaviour is shown not to militate against its value in this connexion. The influence of the glycerin residue in the dried oil film is definite, since freed tung oxyn acids, their soaps, and methyl and ethyl esters are dark coloured, whereas the glycerides are colourless. Analysis of the methyl ester of tung oxyn acids reveals no true hydroxy-acids, and favours the Böeseken and Ravenswaay formulation of elæostearic acid as a conjugated triply-unsaturated substance (B., 1925, 813). A synthetic glyceride of a true hydroxy-acid of the C_{18} group was in no way comparable with an oil film and saponified to a colourless soap. Lines of investigation on many points arising in the present paper are indicated. S. S. Woolf.

Laboratory examination of paints. A. A. Kraeff (Chem. Weekblad, 1927, **24**, 210—219).—Paint films for comparative examination of physical properties are prepared by spreading known quantities over paper

surfaces of definite area previously coated with gum arabic; after drying, the film is removed by soaking in water and washed free from the gum. Chemical examination shows whether the pigment is basic (rust-preventing) or acidic; all acidic pigments accelerate rusting, except chromates, which induce passivity. The permeability of the films by moisture is determined by Gardner's method of closing tubes containing sulphuric acid by films of the paint, and noting the increase of weight after keeping in a damp atmosphere. The permeability is determined chiefly by the oil used and the size of the pigment particles, and by chemical reaction between the oil and the pigment. Tables and graphs of extensions and breaking strains are quoted from work of Gardner and of Nelson, and discussed. The effects of sulphur dioxide and of moisture are examined by exposing films to these reagents in closed vessels, and observing the change in the extensibility. The determination of covering power by means of Pfund's cryptometer is described and some data are recorded, and the use of the Gardner–Holdt flowmeter for determination of brushing consistency is described. Methods of carrying out practical observations on resistance to wind and weather are suggested.

S. I. LEVY.

Physical properties of some grades of whiting. Keeping properties and mobility of paints. H. A. GARDNER (U.S. Paint Manufrs. Assoc., Sept., 1926, Circ. No. 283, 116—123).—The following properties of 10 samples of whiting (natural and precipitated) are tabulated:—fineness, oil absorption, sp. gr., bulk density, alkali number (number of milligrams of potassium hydroxide required by the boiling water extract of 1 g. of sample), colour (whiteness in oil), and times of settling when shaken in water. The settling, alone and when thinned with an equal volume of light petroleum, mobility on keeping, etc. of a series of experimental paints of both exterior and interior types were observed, and it is shown that ready-mixed paints containing 20—30% of precipitated whiting of high oil absorption may show greater mobility than paints of similar composition containing natural whiting of lower oil absorption.

S. S. WOOLF.

Toxic compounds for anti-fouling compositions. H. A. GARDNER (U.S. Paint Manufrs. Assoc., Sept., 1926, Circ. No. 287, 147—149).—A series of submersion tests on anti-fouling paints on metal panels show that red cuprous oxide is the best form of copper for use in such paints, and that copper cyanide is also fairly satisfactory. Mercuric oxide (red or yellow) has excellent toxic qualities, alone or mixed with cuprous oxide, and to a lesser extent with the cyanide.

S. S. WOOLF.

Volatility of acetone–benzene paint and varnish removers. J. M. WEISS (Ind. Eng. Chem., 1927, **19**, 485—487).—The addition of 3—5% of paraffin wax to mixtures of benzene and acetone results in an extraordinary reduction in the rate of evaporation of the solution when exposed to air, due to the formation of a surface film of wax which is almost impervious to benzene vapour. The rates of evaporation of solutions containing wax have been compared with that of the solvent itself at 15·5°, 29·5°, and 37·5°. The reduction in evaporation is greater the higher the m.p. of the wax used. The results indicate that the industrial use of such mixtures as paint and varnish removers is not, under reasonable conditions of ventilation, attended with hazards due to the toxicity of the vapours.

A. B. MANNING.

Physical properties of films prepared from nitrocellulose of low viscosity, and the effect of concentration upon mobility. H. A. GARDNER (U.S. Paint Manufrs. Assoc., Sept., 1926, Circ. No. 284, 124—132).—Lacquers made by dissolving samples of nitrocellulose of extra low viscosity (designated "$\frac{1}{5}$ sec." as against "$\frac{1}{2}$ sec." for the usual low-viscosity type) in a mixture of equal volumes of butyl acetate and toluol were tested, with the addition of plasticisers and pigments, for mobility at various concentrations and for tensile strength and elongation of their films. Promising results were obtained with concentrations of nitrocellulose more than double that possible when "$\frac{1}{2}$ sec." material is used. In the case of a further sample of nitrocellulose of exceedingly low viscosity, giving working solutions containing 45% of nitrocellulose, a danger point in viscosity reduction was indicated, since cracking, checking, and scaling occurred immediately the films dried. The possibility of modifying such products to serve as undercoats is suggested. Details of the falling-sphere method of determining the viscosity of clear lacquers are given.

S. S. WOOLF.

Analysis of cellulose ester varnishes. H. DABISCH (Farben-Ztg., 1927, 32, 1609—1611).—A general scheme for the analysis of nitrocellulose varnish is given, the initial step being the precipitation of the nitrocellulose by pouring the varnish into a suitable amount (determined by a preliminary experiment) of light petroleum, b.p. 35—50°. Addition of a little acetic acid is advantageous if the precipitate remains colloidal. After filtration and washing, solvents are separated from resins and plasticisers by fractional distillation of the filtrate, the fractions being subsequently further analysed. The b.p. and sp. gr. of all commonly occurring solvents are tabulated. In the distillation residue, resins and plasticisers are recognised by saponification, identification of alcohol residues, and re-esterification of the liberated acids according to the Wolff–Scholze method for separating resin and fatty acids. Conclusions as to the nature of the resins present will have been drawn from the presence or absence of cloudiness etc. in the original precipitation of the varnish. Similar details apply for cellulose acetate varnishes, whilst in the case of a blend containing both esters, methyl alcohol is used as precipitating agent, completely bringing down the acetate. The procedure is then as for nitrocellulose varnish.

S. S. WOOLF.

Possible use for hydrocellulose in paints and in lacquers. H. A. GARDNER (U.S. Paint Manufrs. Assoc., Sept., 1926, Circ. No. 282, 113—115).—Hydrocellulose mixed with a high-grade lithopone flat wall paint yields a very thick granular paint that brushes out well and on stippling gives the "pulled" rough effect that has also previously been obtained by the use of plaster of Paris, asbestos fibre, etc. The product obtained by the nitration of dry hydrocellulose is, when washed and

dried, a grey granular material, fairly soluble in lacquer solvents to solutions yielding dark coloured films of apparently good strength. S. S. Woolf.

Examination of turpentine oil used in large-scale chemical industry. C. Austerweil (Chem.-Ztg., 1927, **51**, 249—251).—In the manufacture of synthetic camphor and perfumes (terpin hydrate and terpineol), true turpentine oil (from coniferous balsamic resin) should be used, only α-pinene and nopinene being of use. The oil should be free from water, *e.g.*, there should be no turbidity on treatment with 2 to 3 vols. of benzine; the acid value should not exceed 1, and foreign hydrocarbons, the distillation products of resins, should be absent. The α-pinene and nopinene content is examined thus: the optical rotation should not exceed ± 39° 50′ (pure α-pinene). Distillation after drying over anhydrous sodium sulphate should give a 90% yield of a product having b.p. 153·5—165°/760 mm. The fractions should have d^{15} 0·862—0·874, n^{15} 1·4625—1·4755, and the ratio of the optical rotations for the mercury green (λ=5460) and yellow (λ=5780) lines should be between 1·061 (nopinene) and 1·134 (α-pinene). If the residue of distillation is 10% or more, it is steam-distilled, and the product should have an optical rotation within 10% of that of the original oil. B. Fullman.

Bleaching of shellac. E. R. Watson and H. M. Mulany (J. Indian Chem. Soc., 1926, 3, 255—257).—Shellac when bleached with the minimum quantity of hypochlorite solution (1 pt. of bleaching powder to 6·5 pts. of shellac), precipitated in a granular form by acidification of the alkaline solution in the cold, treated in aqueous suspension with 1 pt. of 10% sodium thiosulphate solution to remove absorbed chlorine, thoroughly washed, and air-dried, can be stored for long periods without any appreciable diminution of its solubility in alcohol. J. W. Baker.

Patents.

Manufacture of carbon black pigments or compositions containing the same. W. H. Whatmough (E.P. 267,625, 22.12.25).—A composite black pigment is produced by precipitating one or more pigments, *e.g.*, Prussian blue, in an aqueous suspension of finely-divided carbon pigment particles. If an oleaginous substance be also present in suspension, the precipitated mass is suitable for use in paints, inks, etc. S. S. Woolf.

Production of titanium pigments. E. C. R. Marks. From J. Irwin and R. H. Monk (E.P. 267,788, 6.8.26).—A titanium pigment substantially free from the higher oxides of titanium is prepared by calcining a finely-ground mixture of precipitated titanium hydroxide with 2—20% of carbonaceous material at 750—900°. S. S. Woolf.

Application of coloured pigments in suspension in gelatin or glue solutions by means of sprays. R. Heinrich (G.P. 435,835, 15.9.25).—The precipitation of the pigment is retarded by using a medium which tends to jellify on cooling. D. Woodroffe.

Potentially reactive liquid coating compositions. E. C. R. Marks. From Bakelite Corp. (E.P. 267,736, 27.4.26).—A phenol is heated under reflux with a fatty oil (preferably tung oil) and a converting agent, *e.g.*, phosphoric acid, anhydrous aluminium chloride, sulphur, oxalic acid, etc., until suitable viscosity is attained when a hardening agent containing methylene groups, *e.g.*, hexamethylenetetramine, is added in amount not exceeding 3% by wt. of the phenol, the necessity for elimination of excess by washing being avoided by this limitation. Reaction proceeds to the formation of a non-reactive, permanently fusible resin which may be used as a varnish base or may be rendered potentially active by dissolving it while hot in a suitable solvent and adding a further quantity of the hardening agent. S. S. Woolf.

Manufacture of aldehyde condensation products. Soc. Chem. Ind. in Basle (E.P. 260,288, 22.10.26. Conv., 22.10.25).—Acraldehyde, alone or in polymerised form, or admixed with other aldehydes, is condensed in one or more stages with carbamide or a derivative thereof in a closed vessel, in the presence of a catalyst, *e.g.*, acetic acid, sulphuric acid, etc. Water, methyl or ethyl alcohol, glycerol, etc. may be added as solvents or diluents, to be separated at the end of the reaction. S. S. Woolf.

Manufacture of linoxyn-like substances. W. O. Hermann and H. Deutsch, Assrs. to Consortium für Elektrochem. Ind. (U.S.P. 1,625,852, 26.4.27. Appl., 13.6.25. Conv., 18.6.24).—See G.P. 434,318; B., 1927, 305.

Impregnating fillers with artificial resins (F.P. 611,264).—See XI.

Tanning condensation products (G.P. 436,446).—See XV.

Photographic process (E.P. 265,769).—See XXI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Influence of butaldehyde-ammonia in vulcanisation. C. A. Hallas and T. J. Drakeley (J.S.C.I., 1927, **46**, 178—179 T).—The effect of the addition of 1% and 2% of butaldehyde-ammonia on the vulcanisation of various rubber mixes was determined. Butaldehyde-ammonia was a powerful accelerator of vulcanisation which was not activated by zinc oxide. In pure rubber-sulphur mixes it acted also as a softening agent, and in mixes containing zinc oxide the softness increased with the time of vulcanisation. In conjunction with diphenylguanidine the softening effect was very marked at the longer times of vulcanisation. The softening effect was not appreciable in a more heavily compounded vulcanisate.

Oxidation of caoutchouc. J. McG. Robertson and J. A. Mair (J.S.C.I., 1927, **46**, 41—49 T).—Purified rubber on oxidation with varying proportions of potassium permanganate in neutral or alkaline solution gives a series of complex, pale yellow or colourless, oxygenated resinous products which are insoluble in acetone, alcohol, or alkalis, but easily soluble in ether, benzene, ethyl acetate, etc. In spite of their inertness these products are probably hydroxy-compounds, analysis indicating mere addition of oxygen without hydration. Formic, acetic, oxalic, and lævulinic acids, also colloidal resin acids are likewise obtained. Oxidation of rubber in a mixture of chloroform and acetic acid by hydrogen

peroxide yields as primary product an alcoholic substance of composition $C_{59}H_{102}O_{16}$, some carbon dioxide also being liberated; the alcoholic substance on oxidation with chromyl chloride gives an aldehyde (capable of yielding a disemicarbazone), whilst on atmospheric oxidation it forms a resin acid apparently with fission of its molecule into two approximately equal parts. Treatment of rubber in carbon tetrachloride with chromyl chloride and decomposition of the primary product with water gives as main products a brown aldehydic resin (oxidisable to a resin acid) and a complex insoluble material containing chromium and chlorine; the latter, which probably arises from polymerisation of the chromyl chloride compound during decomposition, loses its chromium and chlorine when heated with acetic acid or aqueous sodium carbonate, forming an aldehydic resin or a resin acid respectively. D. F. Twiss.

Patents.

Method of preserving [rubber] latex and product obtained thereby. General Rubber Co., Assees. of A. E. Jury and O. H. Smith (E.P. 268,219, 10.9.26. Conv., 8.6.26).—Rubber latex is preserved by the addition of sufficient alkaline phosphate, *e.g.*, trisodium phosphate, to reduce the hydrogen-ion content to below 1×10^{-7}; a bactericide, *e.g.*, formaldehyde, is also introduced. For long storage suitable proportions are 0·5% of formaldehyde and 1·5% of trisodium phosphate. D. F. Twiss.

Production of elastic, waterproof, adherent coatings. H. Quittner (Austr. P. 104,398, 30.12.25).—Paper, fibrous materials, etc. are treated with latex of rubber, gutta-percha, or balata and solutions or emulsions of natural or artificial resins, especially shellac, the latex and the resin dispersion being applied either jointly or successively. The rubber latex may be dilute, concentrated, or vulcanised, or may be compounded and the product subsequently vulcanised. D. F. Twiss.

Waterproof materials (E.P. 266,813).—See V.

XV.—LEATHER; GLUE.

Destructive and preservative effects of neutral salts upon hide substance. II. A. W. Thomas and M. W. Kelly (Ind. Eng. Chem., 1927, 19, 477—480; cf. B., 1926, 23).—Experiments were made on hide powder to determine the action of saturated solutions of sodium, potassium, calcium, and magnesium chlorides, and sodium sulphate, respectively, at 7°, 20—25°, and 37·5°, also of saturated solutions of sodium chloride–sodium sulphate mixtures at 20—25° and 37·5° respectively, and of saturated sodium chloride solutions containing calcium chloride. There was increased hydrolysis with increased temperature in all cases except sodium sulphate. Sodium sulphate solutions showed no hydrolysis at 37·5°, and an increasing amount, though small, down to 7°; this is explained by the greater content of sodium sulphate of a saturated solution at the higher temperature. The different hydrolytic effects of the different salts were not attributable to differences in p_H value. Sodium sulphate is a hide preservative. There was less hydrolysis with various mixtures of sodium chloride and sodium sulphate than with sodium chloride alone. A solution saturated with a salt mixture of 5 mols. of sodium chloride and 1 mol. of sodium sulphate preserved hide powder as well as a saturated sodium sulphate solution. Calcium chloride produced 18% hydrolysis in 7 days; sodium chloride gave 6% at 20—25°, but the admixture of calcium chloride diminished this hydrolysis. The addition of 8% of calcium chloride to sodium chloride renders the latter a better hide preservative. D. Woodroffe.

Action of bates and methods of measuring the enzymatic activity of natural and artificial bates. G. Hugonin (J. Soc. Leather Trades' Chem., 1927, 12, 26—42; cf. B., 1927, 342).—The enzymic activity can be measured by determining the effect on substrates of casein, gelatin, or fibrin respectively, by the Soerensen formaldehyde method or that of Wilson and Merrill (B., 1926, 205). Nine different samples of pancreatin were examined by these methods and by their action on elastin and fats respectively. The Fuld–Gross and Northrop casein methods and the keratose method gave figures of similar order of strength. The activities as determined by their action on collagen and elastin respectively were quite different from the others. The Willstätter and Wohlgemuth methods indicating the activities on fats gave inconcordant results, but they showed that pancreatin possesses lipolytic properties, and further, that the various enzymes in commercial pancreatins are present in very varying proportions. The determination of the activity of a pancreatin should be made on the same product, without using type products. The determination of the quantity of elastin dissolved by a sample of bate can be made under well-defined conditions on the same sample of elastin, but the digestive power of a bate on gelatin, casein, or fibrin is liable to show variations. All the different activities on the various skin components should be determined in order to obtain the practical value of the bate. The Wilson–Merrill method is the best for determining the activity on collagen, elastin, and keratose, and thus the action of a pancreatin or bate on all the constituents of the skin. Willstätter's method is preferable to that of Wohlgemuth for determining the lipolytic activity, since it provides for p_H control and yields easily reproducible results. D. Woodroffe.

Action of iron blacks on leather. D. Woodroffe and D. H. Dew (J. Soc. Leather Trades' Chem., 1927, 12, 42—46).—Pieces of sumac-tanned skiver were treated with solutions containing 5%, 2%, and 1% respectively of ferrous sulphate on the weight of the leather. The soaked leather was dried out, analysed, and kept for 28 months without showing any signs of deterioration. The finished skivers contained 0·97—1·27% of ferric and aluminium oxides. The mere presence of iron in a leather or treatment with iron salts does not appear to cause any deterioration of the leather. Leathers containing iron, which have rotted or weakened, have usually contained free sulphuric acid also. D. Woodroffe.

Commercial gelatins. Their jelly strength, gold number, and hydrogen-ion concentration. P. Serex and M. W. Goodwin (Ind. Eng. Chem., 1927,

19, 473—474).—The jelly strength, gold number, and p_H value were determined on 23 different commercial gelatins of varying grades. The gold numbers showed only slight differences, 0·0085—0·0150. The jelly strength varied from 33 to 339, and the p_H values from 3·99 to 6·70. There was no direct relation between the jelly strength, gold number, and p_H value. A considerable difference in jelly strength between the similar grades of different manufacturers was observed.

D. Woodroffe.

Patents.

Bleaching leather and other products of animal origin. Établ. Reynier and C. Szmukler (F.P. 575,431, 26.12.23).—Wool, hair, pelts, tawed leather, etc. are bleached with dry ozone, and treated with a current of air until the odour of ozone has disappeared.

L. A. Coles.

Bleached and dyed furs. F. B. Dehn. From Stein Fur Dyeing Co., Inc. (E.P. 264,969, 25.11.25).—Fur skins are protected against singeing or burning of the hair or leather and rendered suitable for bleaching by treatment with a ferrous salt, with or without addition of ammonium chloride to retard oxidation of the protective agent. The subsequent bleaching is preferably carried out by hydrogen peroxide or such substances as sodium peroxide or perborate, which generate hydrogen peroxide on treatment with acids. A. Davidson.

Treatment of leather goods. W. Schmidt (G.P. 435,685, 28.4.25).—Articles such as shoes are cleaned with soap solution and then treated with softening agents consisting of water-soluble and emulsifiable oils or fats, such as sulphonated animal, vegetable, or mineral oils.

L. A. Coles.

Treatment of animal tissue and products. F. W. Weber, Assr. to Maywood Chemical Works (U.S.P. 1,621,965, 22.3.27. Appl., 4.4.24. Renewed 6.8.26).—The basic acetates of the rare-earth metals can be employed as tanning agents in place of alum.

T. S. Wheeler.

Manufacture of soluble tanning condensation products from aromatic hydroxy-compounds. A. Kämpf (G.P. 436,446, 21.6.23).—The condensation products derived from phenols or naphthols and natural resins or their constituents in presence of acid catalysts are treated with sulphonating agents, or natural resins or their constituents are treated with aromatic hydroxysulphonic acids in presence of an acid catalyst. Colophony is heated with phenol and sulphuric acid to give a clear solution. After the gradual addition of more sulphuric acid the temperature rises to 70°. The product is soluble in water, and gives a heavy precipitate with gelatin. Similar products can be obtained from colophony and cresol or α- or β-naphthol. In using turpentine and phenol or pinene and α-naphthol, the phenol must be dissolved in the turpentine or its derivatives, condensed with a few drops of sulphuric acid, and sulphonated. The products when dry are brittle resinous substances giving dark yellowish-brown to brownish-green solutions. They are easily precipitated from aqueous solutions by dilute sulphuric acid. The solutions give heavy precipitates with gelatin unless they are neutralised. D. Woodroffe.

Dyeing leather (F.P. 609,904).—See VI.

XVI.—AGRICULTURE.

Fertility of a soil as related to the forms of its iron and manganese. P. H. Brewer and R. H. Carr (Soil Sci., 1927, 23, 165—173).—A type of toxicity present in an Indiana soil was unaffected by dressings of lime or commercial fertilisers. When, however, farmyard manure was added satisfactory yields were obtained. This is attributed to the reduction of some of the ferric iron present to the ferrous state, in which it is more available to plants. The determination of manganese dioxide in soil is discussed. The green colour produced when certain soils are allowed to remain for a long period in contact with alcoholic potassium thiocyanate is considered to be due to the presence of a colloidal manganese compound. It is only developed when the lime requirement of the soil has been sufficiently satisfied by dressings of lime or where insufficient available iron is present. G. W. Robinson.

Outgo of calcium, magnesium, nitrates, and sulphates from high-calcic and high-magnesic limes incorporated in two soil zones. W. H. MacIntire (Soil Sci., 1927, 23, 175—197).—Results are given of lysimetric investigations in Tennessee of the loss in drainage of calcium, magnesium, nitrates, and sulphates, after surface and sub-surface incorporations of calcium hydroxide, burnt dolomite, and a corresponding lime–magnesia mixture, respectively, at a rate equivalent to 0·1% of quicklime reckoned on the soil. The losses of calcium and magnesia were greater with sub-surface than with surface incorporations. Losses of nitrate and sulphate were increased by the dressings.

G. W. Robinson.

Removal of nutrients from subsoil by lucerne. C. E. Millar (Soil Sci., 1927, 23, 261—269).—Exclusion of the top 8 in. of the roots of lucerne plants from the soil by means of glass cylinders did not decrease growth as measured by the weight of air-dry tops. Application of nutrient solution near the tap roots at various depths increased the growth of plants with the root system restricted, but had little effect on normally grown plants. The results indicate that the lower portions of the roots of lucerne are able to absorb nutrients in appreciable quantities. C. T. Gimingham.

Microflora and productivity of leached and non-leached alkali soils. J. E. Greaves (Soil Sci., 1927, 23, 271—302).—The effect of leaching alkali soils on the numbers of micro-organisms and on the biological changes in the soil has been investigated. Both natural and artificial alkali soils were used, the latter being prepared by addition of various proportions of sodium chloride, sulphate, and carbonate to a normal productive soil. Leaching of the normal soil and of all the alkali soils increased the bacterial numbers, in some cases very greatly. Leaching, in many cases, increased ammonification to above that occurring in normal soil. Soil extract and stable manure were very effective in restoring ammonification in alkali soils. Nitrification was very low even after leaching, but was increased by inoculation with soil extract or stable manure. Sodium carbonate reduced nitrification more than the chloride or sulphate. Most of the soils showed nitrogen fixation, which was increased by leaching. Leaching also increased the

crop yields. The author considers that organic manures are essential in reclaiming alkali soils, even where leaching is employed. C. T. Gimingham.

Normal moisture capacity of soils. C. F. Shaw (Soil Sci., 1927, **23**, 303—317).—Experimental evidence indicates that when water is added at the surface to a mass of uniform soil and allowed to percolate downward, a condition of distribution is reached at which the water content at any depth will be approximately the same, except for a region of very rapid decrease in the water content in the lowest layers of the wetted soil. The term "normal moisture capacity" is proposed as an expression denoting the water content of a soil in this condition of uniform distribution; for medium-textured soils it is approximately the same as the moisture equivalent. The author holds that at this point the soil water is essentially in a static condition, and is not free to move under the normal film forces existing in the soil, but it is available for plants. Under these conditions, mulches are of little or no effect in conserving soil moisture. C. T. Gimingham.

The hydrometer as a new and rapid method for determining the collodial content of soils. G. J. Bouyoucos (Soil Sci., 1927, **23**, 319—331).—The percentage of soil material which stays in suspension in water for 15 min. under standardised conditions is equal to the percentage of colloids in the soil, as determined by the heat of wetting method. The agreement is close for nearly all types of soils, the very few exceptions being soils abnormally rich in organic matter not thoroughly decomposed. A quick method for determining the colloidal content of soils is based on this relationship. An amount equivalent to 50 g. of oven-dry soil is dispersed in 1050 c.c. of water by very rapid mechanical stirring for 9 min. in a specially constructed vessel. 5 c.c. of *N*-potassium hydroxide are added, and, after settling for 15 min., the density of the soil suspension is determined by means of a sensitive hydrometer graduated to give grams of dry material per litre directly. The temperature must be taken into account; a change of 1° F. makes a difference of about 0·35% of soil colloids. C. T. Gimingham.

Colorimetric determination of phosphoric acid, particularly in phosphatic fertilisers. C. V. Bordeianu (Ann. sci. Univ. Jassy, 1927, **14**, 353—362).—Citric acid interferes with the colorimetric determination of phosphoric acid by Bell and Doisy's method (A., 1920, ii, 769), but the following reaction may be used. A green ring, becoming blue, appears when a solution containing as little as 0·00005 mg. of phosphoric anhydride in 0·1 c.c. is poured on the surface of a mixture of 2 c.c. of an ammonium molybdate solution prepared as recommended by Fresenius with 1 c.c. of a solution made by adding to 1 g. of *p*-hydroxyphenylglycine 3—4 c.c. of water, then just sufficient nitric acid of *d* 1·12 to dissolve the solid, and making up to 10 c.c. with more water. Hypophosphites and arsenic compounds do not give this reaction, but ammonia and alkaline hydroxides interfere. For colorimetric determination, standard colours are made with a solution of potassium dihydrogen phosphate, which should be of approximately the same concentration with respect to phosphoric acid as the solution to be analysed. The colour requires 15 min. to attain its maximum intensity. The presence of iron does not cause appreciable error if the ratio of phosphoric oxide to ferric oxide is not less than 0·2. Satisfactory results can be obtained with artificial manures, phosphorites, urine, and glycerophosphates. R. Cuthill.

Measure of ionimetric acidity [hydrogen-ion concentration] by inversion of sucrose. Application to complex media: soils. V. Vincent (Compt. rend., 1927, **184**, 338—340).—Phosphate buffer mixtures of p_H 7·12 and 6·98 invert sucrose at 100° as if having p_H 5·20 and 4·80 respectively; this is due to increased dissociation at higher temperatures, or "reserve acidity." The application of this to the study of soils, including those containing calcium carbonate, is discussed.
E. W. Wignall.

Ratio of sulphur trioxide to phosphoric anhydride [in acid phosphate]. Thomas and Howes.—See VII.

Solubility of calcium phosphates in citric acid. Graftiau.—See VII.

Quality of bread from various wheats. Gericke. —See XIX.

Patents.

Inducing germination and growth of seeds and plants. J. Zellweger (U.S.P. 1,625,181, 19.4.27. Appl., 1.8.24).—The apparatus consists of a soil bed on a perforated or porous support with a passageway so arranged that air can pass upwards into the soil. The air is forced through by means of a fan, and moisture is supplied at a high temperature to the fan.
C. T. Gimingham.

Destruction of weeds. A. Rathsack (G.P. 436,405, 15.8.25).—The leaves of weeds are moistened by means of a special spraying device. Materials such as calcium cyanamide, kainit, or anhydrous ferrous sulphate are then applied as a fine powder. G. W. Robinson.

Plant sprays. Farbenfabr. vorm. F. Bayer & Co. (F.P. 596,552, 10.4.25. Conv., 19.4.24).—Plant sprays are prepared consisting of nucleus-substituted mercuric derivatives of organic bases with or without addition of other insecticidal, bactericidal, or fungicidal materials. *p*-Acetoxymercurianiline, its *N*-acetyl derivatives, or the products obtained by the reaction of mercuric acetate with toluidines or other nucleus-substituted anilines or *N*-alkylated aromatic amines, or heterocyclic bases such as pyridine, have a more powerful bactericidal and fungicidal effect than the mercury derivatives of phenols and hydroxy-carboxylic acids, and are more readily miscible with other bactericidal and fungicidal substances. G. W. Robinson.

Fertilisers. Asahi Glass Co., Ltd. (Addn. No. 30,648, 18.2.25, to F.P. 556,211).—The colloidal magnesium silicate of the prior patent is changed into a product containing sodium, which is more stable in the colloidal state, by addition of sodium hydroxide or the use of excess of sodium silicate in its preparation. Potassium or ammonium may be introduced in place of sodium. The magnesium may also be replaced by other metals, such as manganese, and the silicic acid partly or completely replaced by boric acid or phosphoric acid.
G. W. Robinson.

Production of fertiliser from lignite ash. MASCHINENBAU-ANSTALT HUMBOLDT (G.P. 437,515, 20.4.24).—Harmful constituents are rendered innocuous and the active constituents are decomposed by sintering the ash under oxidising conditions; the product is fused in special kilns, and the clinker obtained is ground to a fine powder. L. A. COLES.

Production of phosphate fertiliser. U. WEDGE, Assr. to I. P. THOMAS & SONS Co. (U.S.P. 1,624,195, 12.4.27. Appl., 7.2.23).—A dry mixture of phosphate rock, potassium chloride, and sodium carbonate is heated in a furnace at reaction temperature, when carbon dioxide is liberated and the phosphoric acid content of the rock rendered soluble. W. G. CAREY.

XVII.—SUGARS; STARCHES; GUMS.

Formation and nature of molasses. J. DĚDEK (Chem. Listy, 1927, 21, 96—101, 163—170).—The colloids separated by the dialysis of diffusion juice, of syrup, molasses, caramel, and beet pectins greatly diminish the velocity of crystallisation of sucrose, but in no case does their presence render a saturated sucrose solution non-crystallisable, *i.e.*, colloids are not the cause of the formation of molasses. Their retarding action is due to their adsorption on crystal surfaces, whereby they interfere with the further growth of the crystals. An examination of the saturation solubility of sucrose in solutions also saturated with respect to various salts shows that certain salts may exert a considerable influence on the crystallisation of sugar. Thus lithium chloride lowers the saturation solubility of sucrose to 43·4% of the normal, whilst potassium iodide raises it to 154·2% of the normal.

R. TRUSZKOWSKI.

Determination of sucrose in dried beetroot cossettes. L. EYNON and J. H. LANE (J.S.C.I., 1927, **46**, 177—178 T).—In determining the sucrose content of dried beetroot cossettes by direct polarisation, not less than twice the normal weight, *i.e.*, 52 g. of the material, should be used to insure uniform results. The soluble matter in 52 g. of dried cossettes occupies, on the average, 22·65 c.c., so that the volume of extracting liquid required to make up a total volume of 400 c.c. (a half-normal solution) is $\{400-(22\cdot65+0\cdot52W)\}$, W representing the % of water in the sample. Hence, if the average water content of dried cossettes is assumed to be 6% the amount of liquid required is 374·2 c.c. The procedure consists in heating 52 g. of the sample with 374·2 c.c. of dilute basic lead acetate solution (basic lead acetate solution of d 1·23—1·25 diluted 10- or 20-fold) in a water bath at about 80° for 1 hr., cooling to about 20°, filtering, and polarising the filtrate in a 400 mm. tube; the Ventzke reading represents the % of sucrose in the sample. If the filtrate is not quite brilliant it may be made so by the addition of 0·1 or 0·2 c.c. of acetic acid.

Sugar complexes in wood. FUCHS.—See V.

PATENTS.

Manufacture or recovery of substances from residual liquors of the beet sugar industry. L. MELLERSH-JACKSON. From LARROWE CONSTRUCTION Co. (E.P. 265,831, 17.5.26).—Beet sugar molasses contains glutamic acid probably in the form of an amide. Desaccharified molasses (*e.g.*, Steffen's waste water, distillery slops, or vinasses, etc.) is treated with hydrogen chloride or hydrochloric acid below 70°. A mixture of betaine hydrochloride and inorganic chlorides separates, from which the former is removed by extraction with methyl or ethyl alcohol or concentrated hydrochloric acid. The original mother-liquor is heated at 80—85°, when the glutamic acid derivative is hydrolysed, and glutamic acid hydrochloride crystallises on cooling. The mother-liquor is further concentrated at 50° (or less) for the recovery of more betaine hydrochloride. In an example, Steffen's waste water (d 1·29—1·4) yielded 20% of betaine hydrochloride, 30% of mixed inorganic chlorides (mainly potassium), and 19% of glutamic acid hydrochloride, calculated on the dry matter of the waste water.

B. FULLMAN.

XVIII.—FERMENTATION INDUSTRIES.

Influence of hydrogen-ion concentration on development of colour during wort boiling. W. WINDISCH and P. KOLBACH (Woch. Brau., 1927, **44**, 53—56, 64—67).—In experiments with worts prepared by decoction methods of mashing, as employed in continental brewing, it was found that adjustment of the hydrogen-ion concentration by addition of acid or alkali before boiling influenced profoundly the development of colour during the boiling process. At p_H 6·16 the increase of colour during 2 hrs.' boiling was about 100%, whilst at p_H 5·2 it was only about 30%. At any given p_H value, the increase of colour was proportional to the duration of boiling up to a period of 4 hrs. Hops contribute considerably to the development of colour, but the bitter acids (ether extract) have much less influence in this direction than the portions insoluble in ether. The development of colour during wort boiling is also influenced by the character of the malt and the hops used, and by aeration. It is suggested that in the technical analysis of malts the colour of the standard worts should be determined before and after boiling. J. H. LANE.

Significance of hydrogen-ion concentration in the brewery. H. LÜERS (Woch. Brau., 1927, **44**, 97—103).—The importance now rightly attached to hydrogen-ion concentration in brewing processes should not lead to neglect of titratable acidity. With the use of several indicators showing different p_H values, titration affords useful information regarding the buffer action of worts or extracts. For example, during the malting of barley the p_H value (of cold water extracts) remains almost constant at about 6·0—6·25, but in the course of 4—5 days the titratable acidity towards phenolphthalein (p_H 9) and the titratable alkalinity towards methyl orange (p_H 4·5) both increase to about double their initial values, indicating the formation of soluble buffer substances (phosphates) which stabilise the hydrogen-ion concentration. J. H. LANE.

Standardisation of malt analysis. F. DUCHÁČEK and V. L. ŽILA (Woch. Brau., 1926, **43**, 455—460, 467—472).—Before 1903 there was fairly general uniformity in methods for malt analysis on the Continent. Owing

to the partial or regional adoption of modifications, recommended by a brewing convention at Bonn in 1914 and another at Hanover in 1920, there is now considerable diversity. In most countries the Balling table of densities of sugar solutions has been replaced by the more accurate one compiled by Plato in 1900 as the basis of extract determinations. The new table is calculated for 15°/15° in France and Sweden, for 20°/4° in North Germany, and for 17·5°/17·5° in Bavaria and Czechoslovakia. It indicates amounts of extract about 1% higher than those found by the Balling table. Several test samples of malts were sent by the authors for analysis to 11 laboratories in Germany, and the results are now compared and discussed, and some suggestions are made with a view of ensuring more concordant results. J. H. Lane.

Simplication of work in the analysis of malt and beer. Doemens (Woch. Brau., 1926, 43, 477—481).—Suggestions are made for saving time and work in routine analyses. The sp. gr. of worts can be rapidly and accurately determined by the float method, in which distilled water is added to 200 c.c. of the wort until a bulk, of accurately standardised density, neither floats nor sinks. By noting the temperature of the wort and the volume of water added, the sp. gr. of the wort can be at once ascertained from a table. The method is not so suitable for beers. In the pyknometric weighing of alcoholic distillates barometric variations may cause differences of as much as 5 mg. in the apparent weight of a filled 50 c.c. flask. J. H. Lane.

Head on beer from the physico-chemical point of view. C. Geys (Woch. Brau., 1927, 44, 145—147).—The colloidal particles in beer which contribute most to head-formation and -retention are not the extremely small ones which are electrically charged and disinclined to coagulate, but those of larger size, which are almost or quite neutral, and therefore strongly inclined to coagulation. The latter type of particles tend to accumulate in the surface films, on which they confer stability. During the later stages of brewing, from the cooling of the wort onwards, there is, on the whole, a tendency for the colloidal constituents of the wort to increase in size. This is advantageous for head formation. Excessive coagulation may, however, be detrimental as it removes from the wort many of the particles which would otherwise assist the formation of foam. Long contact of beer with yeast may also be detrimental owing to the peptising action of yeast enzymes on colloids of low dispersion. J. H. Lane.

Aluminium in the brewery. M. von Schwarz (Woch. Brau., 1926, 43, 523—525, 541—545).—Recent experience seems to indicate that aluminium of 99% purity gives better results as a material for brewing vessels than that of lower purity or even that of 99·5%. A defect that occasionally appears a few months after the metal has been rolled is the appearance of very small blisters on the surface, probably due to the presence of minute crystals of a compound $FeAl_3$, formed when the metal is molten and accumulated at the surface in the subsequent rolling. On contact with an electrolyte this compound undergoes corrosion, yielding alumina and hydrogen, but the injury to the metal is usually slight. Aluminium vessels encased in masonry with a layer of pitch between have sometimes corroded owing to the presence of chlorine in the pitch. Unencased vats and tanks are preferred. J. H. Lane.

Empiricism and science in brewing. W. Windisch (Woch. Brau., 1927, 44, 1—5, 13—17, 25—29, 37—41, 49—52, 61—64, 73—76, 85—92).—A personal appraisement of the contributions of science to some of the more important problems of the brewing industry. J. H. Lane.

Bottom- and top-fermentation yeasts. E. Rathke and F. Windisch (Woch. Brau., 1926, 43, 537—541).—Mainly a description of the results obtained by Euler (cf. A., 1926, 1176). J. H. Lane.

Fermentation and ripening of beer. L. Nathan (Woch. Brau., 1926, 43, 585—589, 599—602).—A defence of the Nathan process of brewing against certain criticisms. J. H. Lane.

Saccharification and fermentation of glycogen and starch by maltase-free yeasts. A. Gottschalk (Woch. Brau., 1926, 43, 487—488).—See A., 1926, 544, 759.

Patents.

Denaturising of alcohol. M. Taglietti and S. Hirsch (Swiss P. 115,929, 18.3.25).—As denaturant, rectified oils from ichthyol-shale distillate are used. S. S. Woolf.

Dehydration of alcohol (F.P. 608,062).—See XX.

Treatment of waters for brewing (E.P. 261,708).—See XXIII.

XIX.—FOODS.

Quality of bread from wheats supplied with nitrogen at different stages of growth. W. F. Gericke (Cereal Chem., 1927, 4, 73—86).—Six varieties of wheat received similar applications of sodium nitrate (equal to 100 lb. of nitrogen per acre) at the time of planting and at subsequent growth periods. In some cases addition of nitrogen caused no change in the time of ripening, in others the maturity was delayed. When nitrogen was applied 45 or 75 days after planting, the grain contained the greatest amount of protein. The variation of the quality of the flours, as expressed by the loaf volume and texture of the bread, with the protein content and time of ripening is discussed. F. R. Ennos.

Wheat and flour studies. X. Factors influencing the viscosity of flour–water suspensions. I. Effects of temperature, degree of hydration, and method of manipulation. A. H. Johnson (Cereal Chem., 1927, 4, 87—128).—The viscosity of flour–water suspensions depends on the quantity of electrolytes present and the degree of hydration of the flour constituents. The viscosity is greater the higher the temperature of the water used in the preliminary extraction, since the degree of hydration and the amount of electrolyte removed are increased. Lengthening the period of extraction at lower temperatures and shaking more vigorously have a similar effect. The increase in viscosity is accompanied by a diminution in the resistivity of the wash water, but there is no relationship between the two, owing to the effect of hydration of the

flour, especially at higher temperatures, on the viscosity of the suspensions. Although the electrolyte contained in flour probably consists mainly of dipotassium hydrogen phosphate, sufficient of other electrolytes is present to prevent the calculation of the ash content, by comparison of the resistivities of the decantates with those of this salt. At 50° a change begins to take place in the properties of the flour-water suspensions, as is shown by a sharp increase in the viscosity. By viscosity measurements made after removal of the proteins by pepsin, it is shown that the increase is due mainly to hydration of the proteins, and to a much less extent of the starch. The employment of the viscosimetric method for determining the rate of proteolytic activity in a flour suspension is only possible if the degree of hydration of the flour and the quantity of electrolytes present can be made constant. The method of extraction recommended is to wash with several litres of distilled water at 40° until a maximum viscosity is obtained. F. R. ENNOS.

Determination of milk solids in food products. I. Direct method for milk proteins in cacao products. H. G. WATERMAN and H. A. LEPPER (Ind. Eng. Chem., 1927, **19**, 501—506).—A method is described, directly applicable to milk chocolate, for the separation of casein, lactalbumin, and lactoglobulin as a group from the cacao protein group. Preliminary experiments showed the necessity of removing (*a*) fats by extraction with ether and (*b*) cacao proteins by first extracting with acidified alcohol (85% by vol. of anhydrous ethyl alcohol and 1% by vol. of glacial acetic acid), and then with a weakly acid aqueous medium (5 g. of anhydrous sodium sulphate in 1 litre of 0·015*N*-hydrochloric acid, equivalent to p_H 2·0). The residue is then dehydrated with alcohol followed by ether. Extraction of the milk proteins is performed at p_H 11·6—11·8 by means of a mixture of borate and caustic soda, the borate exerting a buffer action, so that the fall in p_H during the extraction is not more than 0·4—0·6 unit. After centrifuging and filtering, total nitrogen in the extract is determined by the Kjeldahl method. Milk proteins are then determined by selective precipitation with 0·1*N*-sulphuric acid at about p_H 4·5, which point is determined by titration with methyl red as indicator. After removal of the precipitate, non-milk protein nitrogen is determined in the filtrate by the Kjeldahl method. The difference between the two nitrogen figures represents that due to casein, lactalbumin, and lactoglobulin, the conversion factor 6·38 being used. H. J. DOWDEN.

Examination of raw milk by the small plate method. A. M. VAN DEN BURGH (Pharm. Weekblad, 1927, **64**, 417—423).—Comparative figures on the determination of bacteria in milk by the method devised by Frost in 1915 and modified by Clarenburg in 1925 are given, and the method is strongly recommended for the examination of raw milk. With pasteurised milk low results are obtained. S. I. LEVY.

Bacteria causing spoilage of evaporated milk. C. D. KELLY (Trans. Roy. Soc. Canada, 1926, [iii], **20**, V, 387—394).—Three types of spoilage are classified, the bacteria responsible for the changes described being typical forms of *Bacillus cereus*, Frankland, *Bacillus simplex*, Gottheil, and *Bacillus megatherium*, De Bary. A. WORMALL.

Critical solution temperatures and butter analysis. J. WAUTERS (Bull. Soc. chim. Belg., 1927, **36**, 271—276).—Although a normal Crismer value (critical solution temperature in absolute or nearly absolute alcohol) alone is not a proof of absence of adulterants in butter, the sum of the Crismer value and the Reichert-Meissl value is less than the normal 83—84 when cacao butter, neutral lard, or oleomargarine is present. A further confirmation of adulteration is obtained by a determination of insoluble volatile acids (Wauters and Reychler method), which gives higher figures with adulterated than with pure butter. Priority as against Polenske is claimed. C. HOLLINS.

Production of a "caramel" odour and flavour in dairy products by *Streptococcus lactis*, Lister. W. SADLER (Trans. Roy. Soc. Canada, 1926, [iii], **20**, V, 395—409).—Organisms, classified as typical *Streptococcus lactis*, Lister, strains, proved to be responsible for the specific and undesirable flavour of a sample of butter. Remedial and preventive methods, based mainly on sterilisation of the apparatus, are outlined. A. WORMALL.

Gas storage of fruit. F. KIDD, C. WEST, and M. N. KIDD (Dept. Sci. Ind. Res., Food Investigation Special Rep., 1927, No. 30. 87 pp.).—In an attempt to prolong the storage life of fruit by storing in an atmosphere rich in carbon dioxide, with or without temperature control, apples alone being studied, the fruit was stored in closed chambers, restricted ventilation being controlled by opening and closing air ports. The respiratory processes of the apples result in a rise of temperature and an increase in the carbon dioxide content of the atmosphere with a corresponding diminution in that of oxygen. The principal difficulties encountered in storing under restricted ventilation were an increase in humidity to 90—98% and an accumulation of various volatile organic products in the stagnant atmosphere. A high humidity as such has not been proved to be deleterious, but the actual deposition of water on the fruit encourages growth of fungi. The use of oiled-paper wrappers for the apples, or embedding in sawdust or oiled sawdust, removes the ill-effects of the stagnant atmosphere, which otherwise encourages superficial scald. An atmosphere of approximately 10% of carbon dioxide and 11% of oxygen was found to be the most suitable; higher concentrations of carbon dioxide led to a danger of wastage due to brown heart. With this atmosphere, increases in storage life of 50—100% were recorded. Rise in temperature due to self-heating was an important factor, and some form of temperature control was found to be desirable. A temperature of about 10° is believed to be the most suitable. At this temperature an increase in storage life of 100% on a semi-commercial scale has been recorded, but at higher or lower temperatures the efficiency of the method falls off. This is ascribed to variations in the concentration of oxygen and of carbon dioxide in solution in the tissue fluids. At too low temperatures the carbon dioxide concentration in the tissue fluids becomes too high and may lead to brown

heart; at too high temperatures the oxygen content is insufficient to support respiration. The preservation of the colour, firmness, and juiciness of the apples is associated with the prolongation of the storage life. The preserving action of the atmosphere used is found to be proportional to the reduced respiratory activity of the fruit. Discussing overseas transport of fruit, the authors ascribe the varying success of transport in unventilated holds to variations in the extent of accidental ventilation. F. H. GEAKE.

Edible viscera. A. M. WRIGHT and J. C. FORSYTH (J.S.C.I., 1927, **46**, 36—38 T).—Edible viscera and blood represent 24% of the dressed weight of the sheep, 20% of the lamb, and 14% of the steer. Results collected from the work of various other investigators show that vitamins-*A* and -*B* are widely distributed amongst the edible viscera, occurring chiefly in the kidney, liver, and brain, whilst vitamin-*C* also occurs in notable quantity in the kidney and the liver. The results of chemical analyses of the edible viscera from sheep and oxen are tabulated. A canned mixture of hearts and diaphragms marketed as "hearts and skirts" has a very high food value. Blood is a valuable source of albumin. F. H. GEAKE.

Sterilisation of green fodder by a liquid medium. I. C. BRAHM, G. ANDRESEN, and R. PRILLWITZ (Biochem. Z., 1927, **181**, 96—104).—If green fodder be kept in 0·1%—or, better, in 0·2%—hydrochloric acid, the butyric acid fermentation is largely suppressed. Acetic acid fermentation, however, is little affected.
C. R. HARINGTON.

Food dye. JOHNSON and STAUB.—See IV.

PATENTS.

Treatment of condensed or evaporated milk. A. E. VIDAL. From ANDERSON-BARNGROVER MANUF. CO. (E.P. 268,138, 6.4.26).—The hermetically-sealed tins of condensed milk are passed through a steam-heated chamber with a number of divisions, each of which has a higher temperature than the preceding one, the temperature of the last being below that required for sterilisation. After automatic removal of the leaky tins, the remainder are heated in a second chamber at the sterilising temperature (115°), cooled in a third chamber at a pressure above atmospheric, and again passed through a leak detector. F. R. ENNOS.

Manufacture of an invalid food from milk. K. HOEFELMAYR (E.P. 265,910, 23.4.26. Conv., 12.2.26).—Condensed milk, milk powder, or curdled milk is subjected to the action of ultra-violet light for some time. The product has antirachitic properties. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 236,197; B., 1925, 735.] H. ROYAL-DAWSON.

Process for obtaining concentrated vitamin preparations. L. A. AGOPIAN (E.P. 268,655, 24.9.26; cf. E.P. 168,903; B., 1923, 470 A).—Fresh vegetables or fruit are crushed, and the mixture of juice and pulped residue is treated with a solution of lead acetate (80 pts.) and copper acetate (1 pt.). The precipitate, after separation by the filter press, is washed with water, dried, and powdered, the vitamin *A* being extracted with cold light petroleum. The extract is concentrated *in vacuo*, and the vitamin either diluted with a suitable vegetable oil or further concentrated by extraction with 99% methyl alcohol. F. R. ENNOS.

Obtaining extractives from coffee [grounds]. K. VON VIETINGHOFF (E.P. 268,241, 20.10.26).—Coffee grounds are heated with water under a pressure of 8—12 atm. at 160—200°, and the resulting solution, which contains a further 12—18% of aromatic extract, is evaporated if desired. F. R. ENNOS.

Removing the bitter constituents from lupins. GES. FÜR LUPINEN-IND. M.B.H. (G.P. 437,355, 4.5.22. Addn. to G.P. 335,646; B., 1921, 599 A).—Loss of protein is diminished by using solutions containing nitrates, sulphates, or phosphates, instead of salt solution as described previously. L. A. COLES.

Sterilising milk and other products in glass bottles or other containers. F. HUMBEECK (E.P. 268,603, 23.6.26).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Preparation of formaldehyde by catalytic dehydrogenation of methyl alcohol. I. J. C. GHOSH and J. B. BAKSI (J. Indian Chem. Soc., 1926, **3**, 415—430).—The effects of physical aggregation, temperature, and the presence of various promoters on the copper catalyst in the preparation of formaldehyde by the catalytic dehydrogenation of methyl alcohol have been studied. A pure copper catalyst retains its activity for a longer period when the oxide (precipitated from copper acetate, not sulphate) is reduced in the form of small, loose pills or granules (between 10- and 20-mesh) than when it is used in the form of powder. Silver is useless as a promoter, whilst nickel increases the initial activity of the catalyst, which, however, rapidly loses its activity. The best promoters are thorium and cerium oxides. A catalyst prepared from copper acetate containing 0·1% of thorium nitrate at 205° had a space velocity of 900, and yielded approximately 15 g. of formaldehyde per hr. from 16·8 g. of methyl alcohol, and retained its activity unimpaired for an almost indefinite period. With increase in the percentage of thorium or rise of temperature the percentage decomposition of the formaldehyde into carbon monoxide and hydrogen increases. The most efficient catalyst is prepared from copper acetate containing 0·1% of cerous nitrate, which at a reaction temperature of 200° had a space velocity of 1774, and gave an almost quantitative yield of formaldehyde. This catalyst is about twice as efficient as the 0·1% thoria preparation.
J. W. BAKER.

Standards for rectified ethyl alcohol. A. V. RAKOVSKI (Trans. Inst. Pure Chem. Reagents, Moscow, 1927, 26—30).—Standards are suggested for the physical properties and for the permissible impurities in ordinary rectified, double rectified, and purest (prima prima) alcohol, and results of analysis are given for a number of samples received. T. H. POPE.

Preparation of camphor from pinene. H. M. MULANY and E. R. WATSON (J. Indian Chem. Soc., 1926,

3, 253—254).—The preparation of camphor from Indian turpentine may be effected by heating the pinene with 2 pts. of salicylic acid at 110° for 17 hrs. and then gradually raising the temperature to 150° in 11 hrs., a 70·8% yield of bornyl salicylate being obtained. This, on hydrolysis with 5 pts. of 20% alcoholic potassium hydroxide solution for 8—10 hrs., gives borneol in 44% yield. The latter is oxidised to camphor by shaking with 5 pts. of 50% nitric acid for 3 hrs. (yield 50%).

J. W. BAKER.

Manufacture of strychnine and brucine from *Nux vomica*. E. R. WATSON and H. D. SEN (J. Indian Chem. Soc., 1926, **3**, 397—400).—Strychnine may be economically extracted from *Nux vomica* by heating the nuts (41 lb.) with 1·25 times their weight of water for 3 hrs. in an autoclave at 30 lb. pressure, mixing with freshly slaked lime (13 lb.), passing the product through a disintegrator, and extracting with 20 gals. of kerosene oil at 90° for 1 hr. with vigorous stirring in a closed pan. The alkaloid is extracted from the kerosene solution (separated by centrifuging) with 40 lb. of dilute sulphuric acid (containing 240 g. of acid, *d* 1·84), and on concentration of the acid liquor the strychnine sulphate (6·8 oz.) crystallises out. Alternatively, the alkaloid may be precipitated from the acid liquor with soda ash, and the brucine removed by extraction with acetone after drying. The insoluble residue is dissolved in hot, dilute sulphuric acid, from which the strychnine sulphate crystallises out on cooling.

J. W. BAKER.

Biological assay of the specific alkaloid of ergot. J. H. BURN and J. M. ELLIS (Pharm. J., 1927, **118**, 384—387).—The method of Broom and Clark (J. Pharm. Exp. Ther., 1923, **22**, 59) for the biological assay of the specific alkaloid of ergot gives results varying in accuracy from ±10 to ±25%. Examination by this method of extracts of ergots of different origin indicates the following content of alkaloid in the ergot: Portuguese 0·133%, Spanish 0·12—0·067%, Russian 0·005%, and Polish 0·03%. The identity in pharmacological activity of ergotamine and ergotoxin is confirmed.

B. FULLMAN.

Albumose component of *Argentum proteinicum*. C. MANNICH and T. GOLLASCH (Arch. Pharm., 1927, **265**, 96—104).—Nine samples of Protargol of various dates and five other competitive brands all gave the biuret reaction, but only Protargol gave the tryptophan reactions with ferric chloride and formaldehyde and with glyoxylic acid, indicative of the presence of animal albumoses. Protargol also behaved differently towards precipitating agents such as absolute alcohol, 30% acetic acid, and 50% magnesium sulphate solution. Dialysis experiments and precipitation with dialysed ferric chloride solution furnished similar evidence; other brands differed from Protargol in that they contained a less uniform albumose, and more material remained unprecipitated by the iron sol.

S. COFFEY.

Detection of vegetable poisons and drugs in decomposed cadavers. A. BRÜNING and B. KRAFT (Arch. Pharm., 1927, **265**, 106—109).—The remains of the viscera from a case of scopolamine poisoning, which ended fatally in 1923, were kept for three years, during which time decomposition took place. Scopolamine was identified, however, in the decomposed material by the Stas-Otto method.

S. COFFEY.

Determination of total geraniol content in citronella oil and examination of this oil in general. A. RECLAIRE and D. B. SPOELSTRA (Perf. Ess. Oil Rec., 1927, **18**, 130—132).—The formation of citronellal diacetate during the acetylation of citronella oil has been confirmed (cf. de Jong, B., 1919, 232 A), and to the formation of this substance, which is unchanged by heating in a boiling water bath after the addition of water, are ascribed the discrepancies observed in the determination of the "total geraniol" content by different analysts. A method to eliminate the difficulties caused by the presence of citronellal is described. The oil is oximated according to the method of Dupont and Labaune (B., 1924, 926), and in a part of the oximated oil, in which the citronellal is present as citronellaloxime, the nitrogen content is determined by Kjeldahl's method. From this figure the citronellal content is calculated and the geraniol is determined in the oximated oil by acetylation in the ordinary way. The sum of both values is called the "total geraniol" content. Analyses of five citronella oils by different methods and by independent analysts are described. The differences found in the determination of the citronellal, geraniol, and "total geraniol" by the author's methods are much smaller than those given by the recognised methods.

E. H. SHARPLES.

Determination of iodine values. J. BÖESEKEN and E. T. GELBER (Rec. trav. chim., 1927, **46**, 158—171). —In contrast to the ideas of earlier workers the authors take the view that the low or absent iodine values in the case of certain compounds depend not only on the rate of addition of the halogens, but also on subsequent side-reactions of the saturated compound. The authors agree with Ingle (A., 1902, i, 528; 1904, ii, 456) that when the solution of Wijs is employed iodine and chlorine, but not iodine and hydroxyl, are added to the double linking, and they have used this solution to obtain rapidly true iodine values for many substances which contain only one double linking. The so-called negative groups, *e.g.*, carboxyl, phenyl, acetyl, etc., have a retarding influence on the speed of the reaction. In the case of compounds having two (or three) double linkings, it is found that unconjugated systems react rapidly and completely in the normal way, whereas in conjugated systems addition takes place in accordance with the Thiele theory, and the first (or second) addition is rapid, but the final double linking is only slowly saturated. This affords a simple method of distinguishing between conjugated and unconjugated systems, and the authors obtain by Wijs' method iodine values for elæostearic acid which confirm the view that it contains three conjugated double linkings (A., 1925, i, 507). The influence of side-reactions is discussed, and in those compounds in which there is a double linking in the αβ-position to a so-called negative group, the incorrect values are due either to the influence of iodine ions, or to the occurrence of equilibrium reactions, or to the solvent used (dissociating or non-dissociating). Marshall's solution (B., 1900, 213 A) is suitable for the determination of αβ-unsaturated acids. Ingle's theory

of the hydrolysis of the iodochlorides is considered to be incorrect. J. M. Gulland.

Chemical aspects of organic evolution. J. C. Drummond (J.S.C.I., 1927, 46, 161—166 T).

Extraction devices. Clark, also Palkin and Walkins.—See I.

Liquids for mineralogical analyses. Sullivan.—See X.

Turpentine oil in industry. Austerweil.—See XIII.

Patents.

Manufacture of concentrated solutions of formaldehyde or of paraformaldehyde. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 267,768, 18.6.26).—Methyl alcohol, substantially free from water, is catalytically oxidised, and the reaction gases are rapidly cooled so as to condense a mixture of formaldehyde and water vapour, which is then fractionated by means of the uncondensed gases. The resulting concentrated solution of formaldehyde (about 60%) is allowed to solidify to a tallow-like mass, and may be converted into a dry powder by heating *in vacuo*. S. S. Woolf.

Dehydration of alcohol. Soc. Anon. des Distilleries des Deux-Sèvres (F.P. 608,062, 17.12.25. Conv., 18.12.24).—The condensed azeotropic mixture is treated with a concentrated solution of potassium carbonate, magnesium chloride, sodium sulphate, or sodium or potassium acetate. S. S. Woolf.

Preparation of chloro-derivatives of ethane. I. G. Farbenind. A.-G., Assees. of M. Müller-Cunradi (G.P. 436,999, 30.3.21).—Ethane, or a mixture of ethane and ethylene previously reduced, is treated with chlorine in the presence of a catalyst, particularly of active carbon. Equal volumes of ethane and chlorine, led over active carbon at 100—300°, give equal quantities of ethyl chloride and ethylidene dichloride, with small amounts of ααα-trichloroethane. If the mixture of ethylene with a little ethane and other gases adsorbed from coal-gas by active carbon is reduced with the hydrogen corresponding with the ethylene content, and is then passed with an equal volume of chlorine over heated active carbon, there are obtained ethyl chloride and some ethylidene dichloride. Other catalysts, *e.g.*, active silicic acid, may be used. C. Hollins.

Production of alkyl [tetraethyl] compound of lead. G. Calingaert, Assr. to General Motors Corp. (U.S.P. 1,622,233, 22.3.27. Appl., 17.1.24. Renewed 7.11.25. Cf. U.S.P. 1,539,297; B., 1925, 652).—An alloy of lead with 18% of sodium (50 pts.), ethyl iodide (64 pts.), and pyridine (2 pts.) are heated together at 72° under reflux, and water (4 pts.) is added during 2 hrs., at a rate less than that at which the reaction proceeds. The lead tetraethyl, which is formed in satisfactory yield, is removed by distillation with steam. T. S. Wheeler.

Glycol ethers and cellulose ester solvents. Carbide and Carbon Chemicals Corp., Assees. of J. G. Davidson (E.P. 255,406, 21.10.25. Conv., 20.7.25).—As solvents for cellulose esters, mono- and poly-propylene glycol ethers, particularly the methyl, ethyl, and aryl ethers, are used. The monoethers of monopropylene glycol are prepared by heating propylene oxide with the alcohol or phenol in an autoclave at 100—200°, whilst the diethers are obtained from propylene glycol or propylene chlorohydrin by boiling with an alkyl sulphate in presence of alkali. The monoethers of polypropylene glycols, *e.g.*, the ethyl ether of dipropylene glycol, are formed as by-products in the former reaction. The propylene glycol ethers show high solvent power for cellulose esters, the monoethers surpassing the diethers in this respect. They are miscible with most of the common solvents, and are free from disagreeable odour. A. Davidson.

Separation and purification of vanillin. J. Tcherniac (E.P. 268,158, 27.4.26).—Crude vanillin is converted into a somewhat concentrated solution of its sodium bisulphite compound (*e.g.*, 100 pts. of vanillin are dissolved in 328 pts. of a sodium bisulphite solution containing 12·5% of sulphur dioxide), which is then salted out, preferably by the addition of sodium chloride (other salts of sodium, or salts of other metals, *e.g.*, aluminium chloride, may be used); or the solution of the sodium bisulphite compound is treated with the equivalent of a salt (*e.g.*, potassium chloride) which will give an insoluble vanillin–bisulphite compound by double decomposition. The bisulphite compound yields substantially pure vanillin on acidification. B. Fullman.

Preparation of camphor from oil of turpentine. C. Danier (F.P. 579,277, 7.6.23).—Turpentine oil of suitable origin is mixed with 1·5% of caoutchouc as polymerisation agent, and saturated with hydrogen chloride. The almost solid mass is melted with 10—20% of metallic chromium, and after slow cooling is decanted from impurities. The solidified oil is pressed, and yields a synthetic camphor of m.p. equal to that of natural camphor. The process, it is claimed, enables turpentines to be used which are otherwise valueless on account of their mercaptan content. C. Hollins.

Recovery of volatile organic substances from gas mixtures. I. G. Farbenind. A.-G., Assees. of Chem. Fabr. Griesheim-Elektron (E.P. 262,404, 3.11.26. Conv., 3.12.25).—Such solvents as liquid triaryl phosphates or solutions of solid in liquid triaryl phosphates are claimed, *e.g.*, mixtures of isomeric tricresyl phosphates, or the mixed esters obtained according to E.P. 181,835 (B., 1922, 647 A), or solutions of triphenyl phosphate in mixed tricresyl phosphates. The triaryl phosphates have the advantage of low inflammability and volatility, and high solvent power and b.p. B. Fullman.

Preparation of calcium acetylsalicylate. C. R. Jacob (F.P. 609,720, 21.1.26. Conv., 20.10.25).—Carbon dioxide is passed into a mixture of acetylsalicylic acid, calcium carbonate, and just sufficient water to allow the calcium acetylsalicylate to be precipitated as formed. The calcium carbonate is added gradually at the same time as the carbon dioxide. No hydrolysis of the acetyl group occurs. C. Hollins.

Preparation of γ-dialkylamino-α-hydroxy-acids and their derivatives (esters and amides). I. G. Farbenind. A.-G., Assees. of O. Eisleb (G.P. 436,521, 7.12.23).—Dialkylaminoketones are converted into

cyanohydrins which give acids, esters, and amides by appropriate hydrolysis. The *cyanohydrin* of methyl β-dimethylaminoethyl ketone, treated with benzoyl chloride, gives an O-*benzoate*, b.p. 180°/9 mm. (*hydrochloride*, m.p. 206°). When the moist cyanohydrin is dissolved in 37% hydrochloric acid below 35°, heat is developed and a precipitate is formed, which dissolves on further heating, and by evaporation a mixture of ammonium chloride and the *hydrochloride* of γ-*dimethylamino-α-hydroxy-α-methylbutyric acid* is obtained. The crude hydrochloride is converted directly into the *methyl* ester, b.p. 85°/12 mm., *ethyl* ester, b.p. 85—90°/9 mm. or 219—222°/756 mm. (O-*benzoate*, b.p. 180°/9 mm.; O-*benzoate hydrochloride*, m.p. 162°), and *butyl* ester, b.p. 117—118°/9 mm. The *cyanohydrin* of methyl β-diethylaminoethyl ketone yields with alcoholic hydrogen chloride equal amounts of *ethyl* γ-*diethylamino-α-hydroxy-α-methylbutyrate*, b.p. 112—115°/9 mm. or 245—247°/756 mm. (O-*benzoate*, b.p. 195/10 mm.), and γ-*diethylamino-α-hydroxy-α-methylbutyramide*, m.p. 61—62°, b.p. 150—175°/8 mm. (O-*benzoate hydrochloride*, m.p. 167°). The *methyl* ester, b.p. 110°/12 mm. (O-*benzoate hydrochloride*, m.p. 110°), *propyl* ester, b.p. 125°/10 mm. or 259—261°/756 mm. (O-*benzoate*, b.p. 203°/10 mm.; O-*benzoate hydrochloride*, m.p. 114—116°), and *butyl* ester, b.p. 140°/12 mm. (O-*benzoate*, b.p. 215°/12 mm.; O-*benzoate hydrochloride*, m.p. 112°), are described. *Methyl* β-*dimethylamino*iso*propyl ketone*, b.p. 60—64°/20 mm., gives a *cyanohydrin*, from which are obtained *ethyl* γ-*dimethylamino-α-hydroxy-αβ-dimethylbutyrate*, b.p. 95—100°/9 mm. or 223—227°/760 mm. (O-*benzoate*, b.p. 182°/8 mm.). The products have therapeutic interest.

C. Hollins.

Preparation of organic arsenic compounds. Deutche Gold- u. Silber-Scheideanstalt vorm. Roessler (Austr. P. 100,211, 4.3.22).—Halogenated aromatic aldehydes and ketones react with sodium arsenite in aqueous alcoholic solution at 150—200° in the presence or absence of copper or a copper compound. The arsenic acids so formed have trypanocidal action. Examples are *acetophenone*-p-*arsenic acid*, prepared from *p*-bromoacetophenone and sodium arsenite in aqueous alcohol at 160—170°, and 3-*aminoacetophenone*-4-*arsinic acid*, decomp. 230° (*sodium* salt described), prepared from 4-bromo-3-aminoacetophenone, sodium arsenite, and copper powder in aqueous alcohol at 180°.

C. Hollins.

Purification of bisalkylxanthens. A. Rothmann and H. Stein, Assrs. to C. F. Boehringer & Söhne, G.m.b.H. (U.S.P. 1,626,558, 26.4.27. Appl., 12.12.24. Conv., 28.1.24).—See G.P. 431,752; B., 1926, 902.

Products from coal (E.P. 268,006).—See II.

Preparation of olefines (E.P. 248,375).—See II.

Phenols from tar (G.P. 436,444).—See III.

Substances from residue liquors (E.P. 265,831).—See XVII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photochemical studies. IX. Action of certain oxidants on the latent image. A. Reychler (Bull. Soc. chim. Belg., 1927, **36**, 76—78; cf. B., 1926, 515).—An exposed plate is treated for a few minutes with an oxidising solution—chromic acid, acid ferricyanide, mercuric chloride, or acid permanganate. Immediate development gives a very attenuated negative since the insolated grains have been largely destroyed. Exposure to light, followed by development, gives a positive due to insolation of grains previously left unattacked. Removal of the unattacked grains with thiosulphate, followed by development (several hours in full daylight) with a developer made up from sodium sulphite, mercuric bromide, and metol, yields a very vigorous negative, due to development and intensification of the remains of the latent image.

C. Hollins.

Patents.

Mercurial printing surfaces. A. R. Trist (E.P. 265,294, 5.11.25; cf. E.P. 225,928; B., 1925, 152).—A planographic printing plate having ink-retaining and mercurial ink-refusing areas, consists of an iron base on which is electro-deposited first a layer of metal with which mercury can amalgamate, *e.g.*, copper, and then a layer of metal unaffected by mercury to which printing ink will adhere, *e.g.*, nickel. Local resists are applied to the nickel layer to protect the printing areas, and the unprotected parts are then etched away to expose the copper layer and form recesses, which are then filled up with a series of alternately electro-deposited films of two metals, one of which forms a hard amalgam with mercury, such as silver or gold, whilst the other takes a high polish when treated with mercury, such as copper, a film of the latter being outermost; by this means diffusion is reduced to a minimum, and it is possible to arrange that each recess is exactly filled up. The non-printing areas are then treated with mercury.

M. E. Nottage.

Photographic processes [involving synthetic resins from furfuraldehyde, pyrrole, and thiophen]. H. Wade. From Wadsworth Watch Case Co. (E.P. 265,769, 4.2.26).—Resins produced by polymerisation of five-membered monoheterocyclic compounds such as furfuran, pyrrole, thiophen, and their derivatives, or by condensation of furfuraldehyde with ketones and amines, particularly β-naphthylamine, are applied as photosensitive films on suitable surfaces. A sensitiser consisting of a source of halogen, such as iodoform, may be incorporated with the resin. Photographic images can be formed in such media, and are developed by the use of solvents or by selective dyeing. *E.g.*, a mixture of 100 g. of furfuraldehyde and 100 g. of concentrated ammonia solution forms, at room temperature, or under gentle heating if necessary, a resinous condensation product. After drying at 105° it is dissolved in benzene or acetone and applied as a thin film to the surface on which the photographic print is to be made. After exposure to light, the image is developed in a 25% solution of benzene in turpentine.

A. Davidson.

XXII.—EXPLOSIVES; MATCHES.

Detonation wave from solid explosives. W. C. Holmes (J. Franklin Inst., 1927, **203**, 549—559).—The detonation waves of explosives with a detonation velocity of 2000—2500 m./sec. are found to travel with an increase in velocity of 100—200 m. when the wave

leaves the explosives and travels in the surrounding air. In the case of explosives with a high detonation velocity of 5800—6400 m./sec. a decrease in velocity of 1200—1400 m./sec. is observed. It is found that beyond the surface of the explosive the wave travels for a certain distance at a constant velocity and then decreases steadily, and this zone of constant velocity seems to vary with the diameter of the explosive cartridge. With a cartridge of 4 in. diameter the distance is 25 in. compared with 10 in. for a diameter of 1·25 in. as determined by tests on spheres cut out from cartridges of various sizes. Values are calculated for the velocity of the detonation wave in air on the assumption that the wave travels with the velocity of propagation of the gaseous molecules, which is given by the Clausius formula, $v = 29 \cdot 354 \sqrt{T/D}$ where T is the temperature (°Abs.) and D is the density of the gas (air = 1). T is obtained by assuming that the gasification of the explosive takes place adiabatically, and that the Rankine formula is valid. Good agreement between calculated and observed velocities is obtained for blasting gelatin, high-velocity gelatin dynamite 60%, gelatin dynamite 40%, and dynamite 40%. S. BINNING.

PATENT.

Manufacture of detonating compositions for explosive and percussion caps. E. VON HERZ (U.S.P. 1,625,966, 26.4.27. Appl., 21.10.25. Conv., 23.10.24).—See E.P. 241,892; B., 1926, 966.

XXIII.—SANITATION; WATER PURIFICATION.

[Water] chlorination for algæ control. C. COHEN (J. Amer. Water Works Assoc., 1927, 17, 444—455).—In Texas conditions are extremely favourable for algæ multiplication, and the methods of control in general use, which involve treatment with copper sulphate and frequent tank and reservoir cleansings etc., are expensive. Laboratory experiments indicated that algal growth could be inhibited with the application of 1·0 p.p.m. of chlorine. The application of this amount to water entering clean reservoirs and the like was successful in preventing a recurrence of heavy algal growth, but complete freedom from algæ forms was not attained. In another case, where, after nozzle aeration and passage through a filter and prior to distribution, 24 hrs.' settlement of the water was customary, the application of 1·1 p.p.m. of chlorine at the inlet of the settlement tanks, instead of 0·5 p.p.m. (which was usually applied to the settled water for bacterial control), considerably reduced the cost of algæ control, and the treated effluent was as satisfactory from a bacteriological point of view as previously. Further laboratory experiments indicated that dosages of 1·0 and 1·5 p.p.m. of chlorine are effective within periods of 6 and 3 hrs., respectively, and even 0·05 p.p.m. of residual chlorine exercises some effect after 12 hrs.; re-growth of algæ due to seeding and spores does not recur until 8 days from stoppage of chlorine treatment, and that protococcus forms, somewhat resistant to copper sulphate, were susceptible to the action of chlorine. W. T. LOCKETT.

Dissolved oxygen changes during filtration [of water]. W. U. GALLAHER (J. Amer. Water Works Assoc., 1927, 17, 476—480).—There is a gradual increase in the amount of oxygen removed by a filter as the temperature of the applied water increases. Probably most of the oxygen is used up in the biological reactions in the filter bed; whether or not all is used in this manner has not been settled. Temperature may have some relation to air-binding in filters, but is not the sole cause of it. W. T. LOCKETT.

Zeolite softening [of water]. A. S. BEHRMAN (Ind. Eng. Chem., 1927, 19, 445—447).—Artificial zeolites made by fusion and natural products such as bentonite or glauconite are relatively slow in action, and have other disadvantages. A gel zeolite such as "crystalite" is prepared by mixing solutions of sodium silicate and aluminium sulphate under such conditions that no precipitate is formed. The whole mass sets to a gel which may be dried and granulated. Such a zeolite has an economical softening capacity of 14—23 kg. of calcium carbonate per cub. m., or double that of the best of the older materials. This can be considerably increased by a larger consumption of sodium chloride in case of peak loads. The softening plant for a given duty can, therefore, be made much smaller, and water consumption for regeneration and loss of head during treatment are both reduced. With fairly soft waters an upward flow can be used with advantage. In this case loss of head is *nil*, and a rate as high as 10—12 gals./sq. ft./min. can be employed. C. IRWIN.

PATENTS.

Treatment of sewage. T. W. LOVETT (E.P. 266,893, 16.2.26).—Aeration of sewage, prior to and after passing into activated-sludge aeration tanks, is effected by causing a part of the sewage under a head to flow first through a number of vertical tubes each having a part adapted to function as a Venturi tube and below the Venturi throat an air inlet, an enlargement, and a further vertical length, and, secondly, through a vessel which serves as a separator, wherein air ascends to the closed top of the vessel and passes by a conduit to the aeration tanks, while the liquid flows through a lower and open end of the vessel and passes direct to the aeration tanks, or by way of one or more similar Venturi tubes or like conduits. W. T. LOCKETT.

Purifying boiler feed water. A.-G. FÜR STICKSTOFFDÜNGER (E.P. 250,574, 1.4.26. Conv., 11.4.25).—Liberation of oxygen and/or other gases from feed water is effected by forcing the water at a suitable temperature through the nozzle of a water-jet pump into a pipe which, at its upper end, has an enlarged space adjacent to the nozzle. The pipe, at its lower end, is enclosed in a separating chamber, wherein a reduced pressure is maintained, and from which water vapour charged with the oxygen and/or other gases is removed by suction pump, and purified water escapes by a pipe at the base. W. T. LOCKETT.

Treatment of waters for brewing. E. JALOWETZ (E.P. 261,708, 13.8.26).—Either by the addition of gypsum or the removal of the carbonates of calcium and magnesium, the quantity of calcium sulphate in the water is so regulated as to be in exact stoicheiometric proportion to the amount of calcium and magnesium carbonates present. C. RANKEN.

Apparatus for detecting and determining impurities in water (E.P. 268,597).—See I.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 24, 1927

I.—GENERAL; PLANT; MACHINERY.

Mechanical properties of moist granular solids. P. G. Nutting (J. Washington Acad. Sci., 1927, **17**, 185—191).—A theoretical consideration, preliminary to experimental work, of the mechanical properties of moist granular solids with particular reference to moulding sand. It is shown that the forces associated with the merging of the adsorbed water films on small grains at the points of contact are sufficient to account for the order of magnitude of the observed mechanical rigidity, and that the cohesional force increases with the amount of water present up to a maximum, and diminishes to zero corresponding with complete wetting, in qualitative agreement with experiment. R. W. Lunt.

Accuracy of mechanical analysis. C. K. Wentworth (Amer. J. Sci., 1927, **13**, 399—408).—The principal errors in the analysis of sediments by sifting are due to variations in the time of sifting and in the composition of samples divided in different ways, and to the repeated sifting of the same sample. The error due to the second cause is about seven times as great as that due to the third, but of the same order as the time error at 1 hr. of shaking. For comparative purposes, shaking periods of 5—10 min. are sufficient, but for accurate analyses the time must be greatly increased, and should be standardised for any one series. H. F. Gillbe.

Patents.

Rotary furnace. C. B. Wisner (E.P. 246,118, 29.12.25. Conv., 13.1.25).—A tubular rotating furnace which is to be heated outside its full length is provided with alternate stationary and rotating jacket sections, which form with the inner tube of the furnace an annular heating space. The tyres and gearing of the furnace are placed outside the rotating sections, and the joints between the fixed and rotating parts are made of thin overlapping strips of metal in many plies. B. M. Venables.

Furnace [walls]. E. G. Bailey (E.P. 249,560, 22.3.26. Conv., 20.3.25).—A furnace wall is constructed of water tubes faced with refractory material, and by adjusting the thickness of the latter a judicious compromise may be made between low temperature with long life for the refractory material and high temperature with good combustion conditions; in general, the inner surface of the refractory should be just below the fusing point of the ash. Methods of attaching the refractory tiles are described. B. M. Venables.

Furnace settings. Pliibrico Jointless Firebrick Co., Assees. of I. S. Pieters (E.P. 260,953, 17.8.26. Conv., 9.11.25).—A vessel such as an oil still is set over a furnace chamber, which has fireboxes or tunnels constructed within it, from which flame and products of combustion emerge in such a manner as not to impinge directly on the still through side openings, so that flames from one tunnel impinge on those from another, and through top openings with adjustable lids. B. M. Venables.

Furnace. F. I. Pedersen (E.P. 269,397, 6.9.26).—In a furnace comprising a number of superposed combustion chambers or zones, a water heating pipe is embedded in the walls of the lower combustion spaces, and is continued through the flues above the combustion spaces. B. M. Venables.

Furnace. F. A. Fahrenwald (U.S.P. 1,624,043, 12.4.27. Appl., 13.1.26).—The rabble arms of a roasting or other furnace are pivotted at their points of attachment to the driving shaft, and are secured in their working position by comparatively fragile means. B. M. Venables.

Refrigerating machines for obtaining great differences in temperature. Soc. Anon. pour l'Exploit. des Proc. M. Leblanc-Vickers (E.P. 259,921, 29.6.26. Conv., 14.10.25).—A refrigerating machine operating by the evaporation of water or other liquid in a vacuum is arranged with a series of evaporators fixed vertically over each other and a corresponding vertical series of condensers. The evaporator with the highest pressure and temperature is at the top, and the vapour is drawn from it by an ejector or compressor to the lowest condenser, and so on down the series of evaporators and up the series of condensers. The liquid in each series (refrigerant or cooling water, respectively) is sprayed successively in each vessel downwards of the series, and air in the condensers naturally works to the top one, owing to that having the lowest pressure, and can therefore be withdrawn at one point only. B. M. Venables.

Centrifugal grinding mills. B. Scheibaum, Assee. of A. Krause (E.P. 257,947, 3.9.26. Conv., 3.9.25).—A single grinding ball is driven round within a ring, the diameter of the ball being greater than the radius of the ring. Several of these grinding elements may be arranged in line on one shaft, and when thus balanced, the shaft may be driven at a high speed, suitable for direct coupling to electric or other motors. Scraping or deflecting blades may be affixed to the rotor in the spaces between the rings, preferably with a sweep of larger diameter than the rings, also within the rings, at parts of the rotor not occupied by the balls. B. M. Venables.

Crushing and grinding machine. A. C. Hamey (U.S.P. 1,627,506, 3.5.27. Appl., 13.2.26. Conv.,

19.2.25).—A muller is rotated about a vertical shaft, and can also be reciprocated vertically. The feed is central, *e.g.*, down the hollow shaft, and the fixed grinding plate has a central cone surrounded by one or more grooves corresponding to ridges in the muller.

B. M. Venables.

Grinding mill. J. B. C. Scherbaum (E.P. 266,009, 15.2.27. Conv., 15.2.26).—A grinding ring, provided with one or more loose balls within it, is oscillated, *e.g.*, by a crank and pitman, transversely to its axis, about a shaft parallel to its axis. By this means the ball is caused to run round the ring and effect grinding. Several rings may be arranged abreast in one casing with feed chutes and draught of air to remove the ground material.

B. M. Venables.

Grinding mill. E. C. Loesche (E.P. 268,263, 29.12.26).—A shallow bowl is rotated on a vertical axis, and grinding rollers engage with it near its rim. The material is fed to the centre of the bowl, and works upwards and outwards by centrifugal force, passing under the rollers and flying off the edge of the bowl; the coarse is separated from the fine either by a screen surrounding the bowl, or by an air current passing up the annular space between the bowl and the fixed casing. The coarse material, which in either event drops under the bowl, is returned to its upper or grinding side, either by an external elevator or by scoops depending from holes in the bottom of the bowl near the axis.

B. M. Venables.

Grinding and separating apparatus. W. C. Hoover, R. D. Fritz, and E. F. Cahill, Assrs. to B. F. Goodrich Co. (U.S.P. 1,626,466, 26.4.27. Appl., 21.11.23).—A gaseous current—bearing with it the material to be ground—is impelled through an enclosed grinding mill, and through a conduit which leads to the open end of a rotary screen through the meshes of which the gas and fine material pass, while the coarse material is worked back to the open (inlet) end of the screen by internal blades. B. M. Venables.

Rotary mixing machine. J. F. Burn and J. S. Lancaster (E.P. 268,658, 28.9.26).—A bowl for, *e.g.*, a concrete mixer is formed with a stepped bottom or closed end, to promote perfect mixing.

B. M. Venables.

Coal and ore washing installations having a single ascending current. E. Laurent (E.P. 254,693, 15.6.26. Conv., 4.7.25).—A coal-washing apparatus is provided on the supply side with a weir which forms a bed of waste rock, and the rough surface reduces the speed of arrival of the material. On the outlet side it is provided with an adjustable knife-edge for sending a middling product either into the waste or into the coal, or to a separate outlet for the middling alone.

B. M. Venables.

Dryer. Carborundum Co., Ltd., Assees. of C. H. Greenwood (E.P. 260,956, 9.9.26. Conv., 9.11.25).—A rotary dryer which may be fired with oil fuel, for which a high combustion temperature is desirable, is constructed with a tunnel-like combustion chamber of silicon carbide in the bottom of a heating chamber which surrounds the shell of the dryer. The products of combustion, after losing the bulk of their heat in the latter chamber, may be conducted through the dryer.

B. M. Venables.

Drying apparatus. K. Meister (E.P. 268,237, 11.10.26).—A drying apparatus for sticky substances such as brewers' grains comprises a heated feed conveyor, the early part of which is arranged to compress the material and squeeze water out, followed by a trough dryer provided with a false bottom comprising sickle-shaped steam-heated elements, and with air heated by the steam jacket of the feed conveyor. The final drying is effected in a rotary cylinder dryer provided with longitudinal steam pipes inside, over which the material is showered by the rotation of the drum.

B. M. Venables.

Methods of handling material to be sintered. J. E. Greenawalt (E.P. 265,533, 6.4.26. Conv., 5.2.26).—The charge car of a sintering machine of the Greenawalt type is provided with an inclined screen just below its outlet and a chute below the screen, so that the moist charge is separated into coarse and fine, and the coarse material rolls off the screen just ahead of the fine from the chute, and arrives at the sintering grate first, thus forming a first layer of coarse material upon which the fine will rest. B. M. Venables.

Process and apparatus for feeding solids and gases into reaction vessels under pressure. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 268,188, 12.6.26).—A number of auxiliary vessels are connected by pipes to the reaction vessel. An auxiliary vessel is cut off from the pressure, opened, filled with the powdered solid, closed, and a gas pressure rather higher than that in the reaction vessel applied which blows the solid into the reaction vessel. By using the auxiliary vessels in turn the feed may be more or less constant.

B. M. Venables.

Separation of minerals and other substances. T. M. Davidson (E.P. 268,043, 21.9.25 and 27.3.26).—Finely-divided material is fed on to an endless inclined conveyor which is almost wholly submerged in water, and has a transverse vibratory motion imparted to it. The material is preferably fed first to an inclined tray, which is also subjected to vibration and is partially submerged. A partial separation of varying density is thus effected before deposit upon the travelling conveyor. The heavier particles passing upwardly on the conveyor may be finally washed with a water spray before discharge. C. A. King.

Apparatus for separating solids from liquids. W. H. Campbell (E.P. 268,051, 29.12.25).—An apparatus suitable for supporting brewers' grains in the wort, and discharging them after the wort has been drained off, comprises a number of superposed perforated plates, one end of each (except the lowest) is hinged so that it can be raised when charging the grains and used as a chute to distribute the grains over the whole surface of the plates. For discharging the grains the whole set of plates is tilted together and the grain discharged through an opening along one side of the bottom of the tank.

B. M. Venables.

Centrifugal separator. Union A.-G. für Metallind. (E.P. 262,096, 19.11.26. Conv., 30.11.25).—

Liquid to be purified flows through a number of co-axial centrifugal chambers in succession, each one larger in diameter than the preceding, and the outlets (for liquid) from each being at about the smallest radius possible. Near the inner wall of the outermost chamber a co-axial cylindrical filter is arranged, and the liquid passes inwards through this, and is then deflected by helical guides behind the filter up to a final discharge neck or annular chamber which also contains a filter medium. The first filter keeps clean because collected solids are thrown off it by centrifugal force. B. M. Venables.

Method of mounting and driving centrifugal separating apparatus. F. Grimble, M. N. Caird, and E. Coombs (E.P. 269,218, 20.10.25).—The shaft of an underdriven centrifugal machine is provided with rigid radial bearings on each side of the driven worm at the bottom; above that is a universal joint, above that a radial bearing having a certain lateral freedom controlled by springs, and the bowl is attached to the shaft by means that allows a slight lateral oscillation. B. M. Venables.

[Centrifugal] separation of liquid mixtures. J. L. Tufts (U.S.P. 1,623,508, 5.4.27. Appl., 25.4.23).—The mixed liquid is admitted to the lower part and the heavier constituent overflows the top rim of a cage or bowl. The lighter constituent is collected from below an annular skimming ring by means of a scoop-shaped pipe also discharging over the top rim. B. M. Venables.

Apparatus for purifying liquids centrifugally. W. Alexander (E.P. 267,858, 11.12.26).—A mixture of oil and water or other liquids is formed into a vortex within a cylindrical vessel, the vortex being produced by admitting the mixture and/or exhausting the separated water at a suitable inclination produced either by sloping the pipes or by vanes. The separated oil is exhausted at the axis at the upper part, and the conduit may be provided with vanes giving an upward movement to the oil inside the vortex. The inlet for mixture is situated at the circumference near the top, and the outlet for water at some intermediate point (radially) at the bottom. If the last is merely a large central outlet, the vortex will be of the free or Rankine type. B. M. Venables.

Emulsifying apparatus. J. McGougan and J. Hunter (E.P. 267,681, 16.2.26).—The apparatus comprises a hemicylindrical vessel with hermetically closing lid positioned as the diameter of the cylinder and with an internal paddle positioned as a radius. The paddle is provided with a number of small apertures, and when it is oscillated by an external handle the mixture of liquids is forced through the apertures under pressure from side to side. B. M. Venables.

Production of permanent emulsions. P. Lechler (E.P. 254,701, 22.6.26. Conv., 1.7.25).—An emulsion of two liquids, *e.g.*, tar and water, is made in proportions that are known to emulsify easily, and are kept until the liquid has separated into two stable emulsions containing more and less tar. The former is drawn off for market, and the latter is used in the preparation of fresh emulsion. B. M. Venables.

Apparatus for deflocculating and emulsifying. E. A. Butler, Assr. to Colloidal Equipment Corp. (U.S.P. 1,624,037, 12.4.27. Appl., 30.4.25).—An emulsifying chamber has one fixed and one rotating wall, and on each are annular projections forming recesses which are inclined in opposite directions on opposite walls. B. M. Venables.

Manufacture of an emulsion for use in the disincrustation of, or removal of scale from, boilers, hot wells, condensers, and the like. Filtrators, Ltd., and V. Saks (E.P. 268,665, 13.10.25).—In the preparation of an anti-scale emulsion as described in E.P. 176,294 (B., 1922, 280A) by the action of live steam on, *e.g.*, linseed residue, the mucilage or emulsion, instead of discharging direct to the boiler, is discharged through a thermostatic release valve (similar to a steam trap) at atmospheric pressure into the hot well or other desired place. B. M. Venables.

Liquor circulating means for apparatus wherein articles, materials, or substances are treated with liquids, or wherein liquids are mixed or treated. Mellor, Bromley & Co., Ltd., T. C. Bromley, and C. H. Green (E.P. 269,241, 12.1.26).—The dye vat or other vessel to be heated and/or agitated is provided with duplicate sets of air- and steam-jets adapted to swirl the liquid in either direction according to the set of jets used. Valves for changing the direction are automatically operated by a rocking device comprising either a pair of buckets which will overbalance either way when filled by a stream of water above the fulcrum or a paddle wheel in the vat turned by the swirl of the liquid which will lift and overbalance a weighted lever, through the medium of worm gear. B. M. Venables.

Method of treating one liquid with another. S. J. Dickey, Assr. to General Petroleum Corp. of California (U.S.P. 1,625,195, 19.4.27. Appl., 14.5.24).—Oil is treated with a heavier liquid reagent in an apparatus comprising several closed compartments one above the other, in which the oil flows upwards and the reagent downwards; the latter collects on the bottom of each compartment and flows by gravity to the bottom of the next, but the oil is forced through jets in each compartment with sufficient velocity to effect mixing. B. M. Venables.

Fractional distillation. Thermal Industrial & Chemical (T.I.C.) Research Co., Ltd., and W. J. Chadder (E.P. 269,090, 23.11.26).—The vapours from a primary still pass to a dephlegmator, the condensate from which drops to a secondary still underneath, in which a bath of molten metal is maintained at a temperature just below that at which the desired condensate boils. The vapour returns to the dephlegmator, and, together with the remainder of the original vapour, may pass to other dephlegmator-still units for collection of other fractions. B. M. Venables.

Distilling apparatus. J. H. Burlingham, Assr. to Texas Co. (U.S.P. 1,626,223, 26.4.27. Appl., 18.10.21).—The level of the liquid in a still for inflammable liquids is determined by means of an indicator spaced from the still, and which only functions on depression of a trigger, both conducting passages from indicator to still being kept automatically closed. C. O. Harvey.

Separating by vacuum distillation the most volatile constituents of a mixture of liquids. A. SCHMALENBACH (E.P. 269,052, 15.9.26. Conv., 9.6.26. Addn. to E.P. 244,736; B., 1926, 968).—The process described in the original patent is modified by the use of direct steam for distillation and a moderate vacuum.
B. M. VENABLES.

Exchange of heat between gaseous, vapour-like, or liquid materials. MASCHINENFABR. ING. H. SIMMON (E.P. 252,373, 18.5.26. Conv., 19.5.25).—The apparatus comprises a rotor through which one medium is passed while the other medium passes between the rotor and a surrounding casing. The rotor comprises a cylinder with deep ribs perpendicular to the axis, and several methods are described of constructing such a rotor.
B. M. VENABLES.

Apparatus for bringing liquids and gases into contact. [Rectification of liquid air.] L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (E.P. 262,042, 11.5.26. Conv., 28.11.25).—In a rectifying column the trays are constructed to hold an amount of liquid roughly in proportion to the viscosity and surface tension of the gas bubbles at that region; *e.g.*, in a liquid air column the trays in the upper or nitrogen-rich end are made larger and/or deeper than in the lower end, because nitrogen bubbles take so much longer to burst than those of oxygen. B. M. VENABLES.

Apparatus and process for leaching, filtering, and absorbing gases and the like. H. M. STARK (U.S.P. 1,625,831, 26.4.27. Appl., 24.2.23).—A gas filter is constructed with pairs of stamped sheets side by side, with the cut portions so bent that a number of V-shaped capillary spaces are formed between the two plates.
B. M. VENABLES.

Process and apparatus for treating liquids with purifying and decolorising agents. J. N. A. SAUER (U.S.P. 1,627,343, 3.5.27. Appl., 25.5.23. Conv., 26.5.22).—See E.P. 198,366; B., 1924, 321.

Furnaces [for preheating etc.]. A. SMALLWOOD and J. FALLON (E.P. 266,923, 6.4.26. Addn. to E.P. 255,938; B., 1926, 792).

[Device for removing the lids of] furnaces. BRITISH THOMSON-HOUSTON CO., LTD., and A. N. OTIS (E.P. 255,479, 16.7.26. Conv., 16.7.25).

Heating or cooling viscous liquids. GRISCOM-RUSSELL CO., Assees. of R. C. JONES (E.P. 256,579, 21.6.26. Conv., 5.8.25).

[Basket cover for] hydro-extractors or centrifugal machines. A. F. DUNSMORE (E.P. 268,701, 30.12.26).

Process and apparatus for charging a liquid with radioactive emanations or gases. A. LEPAPE and E. LEVOUX (E.P. 267,764, 3.6.26).

Flotation apparatus (U.S.P. 1,624,559).—See X.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Primary decomposition of coal. I. Temperature of initial decomposition. J. G. KING and R. E. WILLGRESS (Dep. Sci. Ind. Res., Fuel Res., 1927, Tech. Paper No. 16. 19 pp.).—A series of coal samples (20 g. each), from peat to anthracite, of 10—20-mesh size, were mixed with freshly-ignited sand (40 g.) of the same size and heated in a vertical tube placed in an electric furnace and connected with a cooled U-tube. The temperature of this mixture was raised 10° per hr., while an inert gas was passed through the tube, and the appearance of moisture and oil was observed. When the inert gas was nitrogen the temperature of initial appearance of oil from peat was 180°, from lignite 250°, and from bituminous coal 215° (for caking coal) to 240° (for non-caking coal). Substantially the same figures were obtained when using carbon dioxide or hydrogen, or working *in vacuo*, but no definite value was obtainable with anthracite. Decomposition continued for some hours at least if the coal was maintained at the critical temperature. With bituminous coals the temperature of initial decomposition appears to increase with increasing oxygen content and decreasing carbon content. The evolution of water from coal in the absence of air is a gradual process, but with certain coals there are temperatures at which the rate of evolution is accelerated, the first acceleration of moisture evolution coinciding, for bituminous coals, with the appearance of oil vapour. The temperatures of initial decomposition here recorded are much lower than those found by previous investigators.
W. T. K. BRAUNHOLTZ.

Inflammation of coal dusts: effect of the chemical composition of the dust. T. N. MASON and R. V. WHEELER (Safety in Mines Res. Bd., 1927, Paper No. 33. 20 pp.).—Mixtures of coal dust and fuller's earth, both containing about 85% passing through 200-mesh, were spread on the floor of a gallery 38 in. in diameter and 308 ft. long, closed at one end. Ignition of the dust was effected by a cannon, aided by an "impetus tube." The inflammability of the dusts of a series of British bituminous coals of equal fineness, measured by the amount of incombustible matter which must be present in the mixed dust in order to prevent continued propagation of flame, was related to the content of volatile matter in the coal (ash-free and dry basis), the dusts from coals with a high content of volatile matter tending to be more readily inflammable. It is not suggested, however, that the content of volatile matter directly determines the inflammability of the coal dust. No difference was found between the inflammability limits of fresh and weathered dusts from a given coal. The effects on flame propagation of the concentration of dust in the gallery and "impetus tube" and of varying the cannon charge were also studied.
W. T. K. BRAUNHOLTZ.

Upper Silesian blast-furnace coke. F. BÖNNEMANN (Glückauf, 1926, **62**, 1551—1557; Chem. Zentr., 1927, I, 543).—A systematic study has been made of the coking properties of coals from various seams, none of which, by itself, yields a good coke. By determining the most suitable particle size, width of oven, temperature, and mixture ratios, it has been possible to produce a coke which works smoothly in the blast furnace, giving a 60% increase in the yield of iron and an improvement also in the quality of the latter. A. B. MANNING.

Purification of town gas in relation to corrosion. J. Parker (Gas J., 1927, **178**, 361—362).—Corrosion inside gas mains is mainly attributed to the condensation of water vapour from the gas in the presence of oxygen, carbon dioxide, etc. This can be prevented if the saturation of the gas is reduced below 40% by washing with a strong solution of calcium chloride in a "brush" horizontal rotary washer. The solution is continuously circulated through the washer *via* a cooler to extract the heat evolved in the washing process, the diluted solution being reconcentrated by means of exhaust steam from the engine driving the washer. The cost of the process when operated in conjunction with that of naphthalene extraction does not exceed 0·2d. per 1000 cub. ft. of gas treated. Tests carried out on the dried gas have given no indication of the presence of iron carbonyl. H. D. Greenwood.

Apparatus for determining the content of solvent vapours in gases, particularly of benzene hydrocarbons and gas benzine from the carbonisation of coal. A. Weindel (Brennstoff-Chem., 1927, **8**, 136—138).—The tin adsorption tube containing active charcoal is inserted into a thick-walled aluminium cylinder heated by gas. Steam is superheated by passing, on its way to the tin tube, through a tube bored out of the wall of the cylinder, in which also a thermometer is inserted. The temperature of the aluminium cylinder is raised to 350° (corresponding to a temperature of 320—330° in the tin tube), and the expulsion of the solvent is complete in 25—35 min. Only 100—150 g. of steam are required. W. T. K. Braunholtz.

Products from the cracking of lignite producer tar oils and petroleum gas oils. G. Varga and A. Erdély (Brennstoff-Chem., 1927, **8**, 133—136).—One litre of oil was rapidly heated in an autoclave till the internal pressure reached a given figure, and the cracked products were withdrawn while that pressure was maintained. The yields of oils boiling up to 200° were about 45—57% from various gas oils, and 32—47% and 28—32% from refined and crude lignite producer tar oils, respectively. At 10—20 atm. pressure the lignite producer tar oils began to decompose at 370—390°, and the maximum yield of benzine was obtained at about 420°, whilst above this temperature coke and gas formation predominated. The corresponding temperatures for the Roumanian, Russian, and Texas gas oils examined lay about 20° higher. With gradually increasing cracking temperature the sp. gr. of the benzine from the lignite oils first sank and then rose. There was no material difference as regards boiling range, sp. gr., and solubility in 100% sulphuric acid between the benzine obtained from petroleum and that from refined lignite producer tar oils. W. T. K. Braunholtz.

Utilisation of gas coke. E. W. Smith (J. Soc. Arts, 1927, **75**, 572—596).

Spalling of refractories. Green and Dale.—See VIII.

Use of super-refractories in gas manufacture. Knollman.—See VIII.

Coke-oven construction and refractories. Vickers and Green.—See VIII.

Discoloration of glass by coal gas. Offe.—See VIII.

Gallium in flue-dust. Ramage.—See X.

Patents.

Decomposition of coal and hydrocarbons by heating with hydrogen under pressure. Soc. Internat. des Combustibles Liquides (F.P. 606,189 and 606,191, 12.2.25).—(a) The reaction is carried out in two stages, the material being hydrogenated in the first and decomposed into separate fractions in the second. By heating a mixture of powdered bituminous coal and tar oils, in the proportion 2 : 1, directly at 440—475° in the presence of hydrogen at 100 atm. pressure, no liquid product is obtained, but by heating first to 420° within 24 hrs., and then at 440° for 2 hrs., a 56·9% yield of oil boiling to 330° is obtained. The amount of hydrogen taken up increases with the pressure, whilst the amount of decomposition varies inversely with the pressure. The yield and quality of the low-boiling fraction of the oil are better the greater the hydrogen absorption during the first stage. When a mixture of equal parts of coal and coal-tar residues is heated in the autoclave at 210° it becomes liquid, free carbon and ash settling out, but the yield of oil of low b.p. is small. If, however, this product is again hydrogenated after the addition of 5% of alkaline iron oxide a good yield of oil boiling to 230° is obtained. (b) The starting materials are pumped under pressure into the reaction chamber, which is provided with stirrers. The liquid reaction product can be discharged through a tube which dips below its surface, into a container fitted with a valve. The separation of the solid, liquid, and gaseous products is carried out under normal pressure. A. B. Manning.

Production of fuel. C. J. Greenstreet, Assr. to American Coalinoil Corp. (U.S.P. 1,623,241, 5.4.27. Appl., 13.9.22).—Coal is ground and agitated with a mixture of water and fuel oil of density greater than 1·18. The impurities are thus separated from the coal, which is dispersed in an emulsion of oil and water. The product, which is of value as a fuel, can be rendered more stable by blowing it with air to oxidise and thicken the oil. T. S. Wheeler.

Production of a binding fuel material. T. Nagel (U.S.P. 1,626,208, 26.4.27. Appl., 24.5.24).—Pulverised fuel is incorporated in suitable proportions with a mixture of alcohol slops and sulphite liquor, then pressed, and heated at 250—500°, to convert the mixture into a hard insoluble smokeless binder. H. Royal-Dawson.

[Fuel] pulveriser. Syracuse Pulverizer Corp., Assees. of A. J. Briggs (E.P. 258,896, 6.10.25. Conv., 8.8.25. Cf. E.P. 256,562; B., 1926, 999).—A pulveriser for fuel which draws sufficient air for combustion, and can be placed close to the furnace where the fuel is burnt, is constructed with a disintegrator of the hammer type surrounded by one of the intercalating tooth type, the powdered fuel being drawn off by a fan on the same shaft. Control devices for air and fuel are provided. B. M. Venables.

Gas generating apparatus. F. Umpleby (E.P. 269,269, 29.1.26).—The generator consists of a chamber of refractory material and having uneven (grooved) walls.

The gasification is effected by the heat generated by surface combustion on the walls. The air or oxygen and the material to be gasified are admitted to the chamber by regulatable valves, and the gas being generated can be modified by the admission of steam, carbon dioxide, etc. Steam is also admitted in the form of a screen through which the ash or residue from gasification must fall and is cooled. Catalysts may be used, also a rotary motion imparted to the fuel. The direction of flow of the generator and burning gases may be reversed in order to assist the separation of the heavier particles of ash as residue. The fuel may be introduced in the form of a spray. A suitable device is provided for the discharge of may also be made of a heat-resisting nickel alloy or nickel itself. A series of chambers for a graduated gasification the ash. The reaction chamber, valves, and other parts process may be provided. The gas may be generated from the volatile matter of carbonaceous material, and the fine coke-like residue fed to the furnace. The plant may operate in conjunction with a pulverised fuel or briquetting plant. R. A. A. TAYLOR.

Generation of combustible gas. J. E. HACKFORD, and HAKOL, LTD. (E.P. 268,899, 16.11.26).—The process described in E.P. 217,613 (B., 1924, 703) is modified in that the air admitted to the chamber is directed by several different suitable means round the walls of the chamber so that the fuel is prevented from coming into contact with the walls and depositing carbon on them. R. A. A. TAYLOR.

Manufacture of water-gas. H. NIELSEN and B. LAING (E.P. 269,234, 6.1.26).—The endothermic nature of the water-gas reaction is counterbalanced by the admission with the steam (which may be superheated) of a quantity of more or less inert gas admixed with carbon dioxide. The gas and steam may be heated in a chequer brickwork chamber and then brought into a second chamber to react with coarsely pulverised semi-coke. The sensible heat of the water-gas made can be used to effect the carbonisation of coal. The material to be carbonised is allowed to fall down a vertical space between superposed shelves.
R. A. A. TAYLOR.

Coke ovens. N. V. SILICA EN OVENBOUW MIJ., Assees. of C. OTTO & Co., G.M.B.H. (E.P. 259,968, 13.10.26. Conv., 14.10.25).—A vertical chamber oven has two regenerators on one side only. They are separated by the chimney-flue common to both, and are connected with the heating-wall on the one side and with the flue, gas-main, or air on the other. The products of combustion thus follow a C-shaped path. The air and gas may be distributed so as to meet only at the top or bottom of the heating-flues. Mains for the distribution of rich or lean gas or both may be provided, together with suitable regulation of the openings from the distributing-flue. R. A. A. TAYLOR.

[Lampblack from natural] gas. STANDARD DEVELOPMENT CO. From J. P. GARNER (Can.P. 261,588, 26.5.24).—Natural gas is cracked to produce lampblack, giving at the same time a low-grade gas. The latter is enriched by the addition of a hydrocarbon which is not liquid at the ordinary temperature, and which suffices to bring the calorific value up to that required for ordinary use.
A. B. MANNING.

Acetylene and lampblack from liquid hydrocarbons, *e.g.*, petroleum. C. LONGHI (F.P. 612,036, 2.3.26. Conv., 7.3., and 21.9.25; cf. E.P. 248,830; B., 1926, 525).—The known reaction whereby acetylene is produced by the action of the electric arc under the surface of a liquid hydrocarbon rapidly ceases owing to the formation of a layer of vapour round the electrodes. This is avoided if the arc is extinguished immediately after its formation and re-formed at once in another place. Thus the oil is contained in a cylindrical vessel in which a disc rotates rapidly in such a manner that arcs are continually being formed and broken between a large number of points serving as electrodes on the disc and corresponding electrodes on the walls of the vessel. The same result is attained by the use of two discs without points which rotate in opposite directions. A. B. MANNING.

Treating oils. G. EGLOFF and W. R. HOWARD, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,627,164, 3.5.27. Appl., 8.6.21. Renewed 5.2.27).—Heated oil is passed into an enlarged reaction chamber through a pipe, the open end of which is situated at the end of the chamber remote from the point of entry of the pipe. This end of the chamber carries a downward extension, and the vapours are withdrawn at a point remote therefrom. C. O. HARVEY.

Treating impure crude oil. J. F. WRIGHT (U.S.P. 1,627,072, 3.5.27. Appl., 26.1.24).—The oil is sprayed against a rotating member and comes in contact with a spray of hot water. Separation is effected in vertical chambers. C. O. HARVEY.

Treatment of petroleum emulsions. C. C. AVERILL, Assr. to W. S. BARNICKEL & CO. (U.S.P. 1,617,737—1,617,741, 15.2.27. Appl., [A—E], 2.9.24, 12.10.25, 21.11.25, 3.5.26, 26.6.26).—(A—C) Oil-water emulsions are broken by emulsification with water followed by chemical demulsification. (D) Water-in-oil emulsions are homogenised and subsequently subjected to electrical dehydrating action. (E) Water-in-oil emulsions are treated with a chemical demulsifying agent, homogenised to reduce the size and increase the uniformity of the water droplets, and dehydrated electrically. C. O. HARVEY.

Purification of benzol, petrol, and the like. K. COX and P. J. MCDERMOTT (E.P. 269,242, 12.1.26).—The oil or spirit is heated with ferric sulphate and, if necessary, with a micro-porous material, and the distillate is then treated with alkali to neutralise its acidity.
R. A. A. TAYLOR.

Refining oils. H. O. PARKER, Assr. to MATHIESON ALKALI WORKS (U.S.P. 1,627,338, 3.5.27. Appl., 18.6.25).—The oil is passed through a succession of mixing and settling operations, an alkaline aqueous solution of hypochlorite being supplied to one of the mixing chambers, allowed to settle out in the succeeding settling chamber, and then passed back to similar mixing and settling chambers preceding those to which the aqueous solution is first supplied. C. O. HARVEY.

Purification of waste acid from crude oil refining. W. DEMANN (G.P. 436,242, 10.9.25).—The waste acid is warmed or diluted with water, mixed with heavy benzine, tetralin, or other hydrocarbon of aliphatic

or polymethylene character, and the mixture then separated by centrifuging. A. B. Manning.

Purifying hydrocarbon oils. J. C. Morrell and S. Comay, Assrs. to Universal Oil Products Co. (U.S.P. 1,627,055, 3.5.27. Appl., 22.6.25).—The oil is treated with a solution of copper hypochlorite. F. G. Crosse.

Distilling hydrocarbon material. E. M. Clark, Assr. to Standard Development Co. (U.S.P. 1,625,984, 26.4.27. Appl., 17.1.21).—The pitch residues resulting from the steam-distillation of hydrocarbon oils are heated, while supplying steam, until still wax distils over, when the distillation is continued without steam to the coking stage. C. O. Harvey.

Reactivation of desulphurising agents for hydrocarbon oils. J. B. Rather and F. S. Shepard, Assrs. to Standard Oil Co. of New York (U.S.P. 1,622,671, 29.3.27. Appl., 16.4.24. Renewed 12.8.26).—Copper oxide which has been employed for the desulphurisation of hydrocarbon vapours is regenerated by treatment with superheated steam to remove adhering oil, and then with air at 260° to convert the sulphide present into oxide. T. S. Wheeler.

Removal of sulphuretted hydrogen from gas. E. W. Smith and T. C. Finlayson, Assrs. to Woodall-Duckham, Ltd. (U.S.P. 1,628,477, 10.3.27. Appl., 14.11.25. Conv., 10.12.24).—See E.P. 245,575; B., 1926, 230.

Destructive distillation of solid carbonaceous materials. P. Dvorkovitz (U.S.P. 1,627,321, 3.5.27. Appl., 16.10.22. Conv., 17.11.21).—See E.P. 192,816; B., 1923, 301 a.

Free-discharge separators of plant for washing coal and other minerals by means of liquid streams. A. France (E.P. 269,017, 28.6.26).

Separation of liquids and solids of low melting point (G.P. 437,482).—See I.

Fractional distillation (E.P. 269,090).—See I.

Furnace settings (E.P. 260,953).—See I.

Separation of minerals etc. (E.P. 268,043).—See I.

Coal washing installation (E.P. 254,693).—See I.

Washing minerals (E.P. 268,262).—See X.

Fatty acids from hydrocarbons (U.S.P. 1,627,452).—See XII.

Manufacture of methyl alcohol and hydrocarbons (E.P. 247,178).—See XX.

III.—TAR AND TAR PRODUCTS.

Cracking of lignite producer tar oils. Varga and Erdély.—See II.

Patents.

Permanent emulsions (E.P. 254,701).—See I.

IV.—DYESTUFFS AND INTERMEDIATES.

Dyes in wine. Valentini.—See XVIII.

Patents.

Production of 1-naphthol-8-carboxylic acid. R. Herz and F. Schulte, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,623,678, 5.4.27. Appl., 12.7.26. Conv., 29.11.24).—1-Cyanonaphthalene-8-sulphonic acid is hydrolysed with 60% sulphuric acid at 70° to 8-*sulpho*-1-*naphthoic inner anhydride*, m.p. 151—152°, which product on fusion with potassium hydroxide at 200° yields 1-naphthol-8-carboxylic acid. T. S. Wheeler.

Manufacture of arylamides of o-hydroxycarboxylic acids and of intermediate products for azo dyes. British Synthetics, Ltd., and E. B. Higgins (E.P. 268,877, 7.1.26).—Quantitative yields of arylamides are obtained by adding an arylamine gradually to an *o*-hydroxycarboxyl chloride stirred in a solvent or diluent (benzene) maintained at 60°. Hydrogen chloride is evolved and only 1 mol. of the amine is necessary. Examples are 2 : 3-hydroxynaphthoic anilide and β-naphthylamide. C. Hollins.

Preparation of stable diazo compounds. I. G. Farbenind. A.-G. (F.P. 610,261, 29.1.26).—Solid aromatic diazo compounds, with the exception of those derived from the aryl or aralkyl ethers of *o*-aminophenols, are mixed with metallic salts of aromatic sulphonic acids, with or without the addition of copper salts, aluminium salts, or alkali chromates. Numerous examples are given, the stabilising compounds being: technical sodium naphthalene-1 : 6-disulphonate; the same with anhydrous aluminium sulphate; sodium naphthalene-tri- and -tetra-sulphonates from naphthalene and 4 mols. of sulphuric acid monohydrate; sodium benzenesulphonate and tetralinsulphonate; sodium naphthalene-2 : 7-disulphonate and crystallised copper sulphate. A moist 72% paste of *p*-diazobenzenesulphonic acid and sodium naphthalene-1 : 5-disulphonate is dried without difficulty at 40—50°. In some examples diazo salts already stabilised as zinc chloride double salts or in the form of their *p*-chlorobenzenesulphonates etc. are mixed with aromatic sulphonates. C. Hollins.

Preparation of stable diazo compounds. Chem. Fabr. Griesheim-Elektron (F.P. 600,311, 3.7.25).—Aqueous solutions of nuclear substituted benzenediazonium chlorides, excepting sulphonic acids and azo derivatives, give stable zinc chloride double salts, $2RN_2Cl,ZnCl_2$, which may be salted out with sodium chloride, and dried either at 50—70° or by admixture with partly anhydrous aluminium sulphate etc. C. Hollins.

Production of a [vat] dye [of the anthraquinone series]. W. G. Woodcock, H. A. E. Drescher, E. G. Beckett, J. Thomas, and Scottish Dyes, Ltd. (E.P. 268,537, 3.10.25).—A blue vat dye, brighter in shade and faster to chlorine than that obtained from the product of reduction of 2 : 4-dichloro-1-aminoanthraquinone, is prepared by the condensation of 2-chloro-1-aminoanthraquinone, m.p. 196°, obtained by the method of E.P. 264,916 (B., 1927, 246) and purified by precipitation as *sulphate* from 70% sulphuric acid. The condensation takes place in presence of sodium acetate (etc.) and copper (etc.) in hot nitrobenzene, naphthalene, etc. C. Hollins.

Manufacture of intermediates of the anthraquinone series. British Dyestuffs Corp., Ltd., and W. W. Tatum (E. P. 268,891, 11.1.26).—The preparation of 1 : 4-diamino- or 1 : 4-dialkyldiamino-anthraquinones in the form of their leuco-compounds is performed in a

single operation by treating a 1 : 4-dihydroxy- or 1-amino-4-hydroxy-anthraquinone with ammonia or an alkylamine in the presence of sodium hyposulphite and water, in an autoclave, if desired, or in an open vessel. Quinizarin, sodium hyposulphite, and aqueous ammonia at 70—90° give leuco-1 : 4-diaminoanthraquinone. Similarly leuco-1 : 4-dimethyldiaminoanthraquinone is prepared from 1-amino-4-hydroxyanthraquinone, aqueous methylamine, and hyposulphite at 30—80°; leuco-5-amino-1 : 4-dimethyldiamino-8-hydroxyanthraquinone from diaminoanthrarufin; leuco-1 : 4-dimethyldiamino-7 : 8-dihydroxyanthraquinone from 1 : 4 : 7 : 8-tetrahydroxyanthraquinone. The last two products after oxidation give greenish-blue dyes for acetate silk. C. HOLLINS.

Preparation of nitriles of the benzanthrone series. M. P. SCHMIDT and W. NEUGEBAUER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,628,280, 10.5.27. Appl., 23.2.26. Conv., 17.11.24).—See E.P. 243,026; B, 1927, 101.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Transverse sections of artificial silk. Y. KAMI and S. NAKASHIMA (J. Cellulose Inst., Tokyo, 1927, **3**, 87—94).—Photography of cross-sections of threads is used as the safest method of controlling artificial silk manufacture and for judging the nature of the product. Comparisons are shown between sections and the coagulation of the cellulose derivative solution (*i.e.*, composition of the spinning bath, bath temperature, drying of the raw silk, etc.). Four methods of preparing the sections are described and compared, and the effects of these methods in showing differences between various kinds of artificial silk are illustrated. Differences in the degree of swelling of sections prepared by the four methods are shown by measurements of their area, and the ratio area (sq. microns) : denier of a single fibre is termed the softness factor of the silk. B. P. RIDGE.

PATENTS.

Cooking vegetable fibre. S. D. WELLS (U.S.P. 1,626,171, 26.4.27. Appl., 29.6.26).—Fibrous material is softened by treatment with a solution of sodium carbonate and sulphur. H. ROYAL-DAWSON.

Manufacture of a cellulose-fibre product treated with a size embodying soya-bean flour. G. DAVIDSON, H. F. RIPPEY, C. N. CONE, I. F. LAUCKS, and H. P. BANKS, Assrs. to I. F. LAUCKS INC. (U.S.P. 1,622,496, 29.3.27. Appl., 3.5.26).—Soya-bean flour dispersed in water, with or without addition of weakly alkaline salts, is employed as a size for paper or the like. T. S. WHEELER.

Spinning process. H. L. J. CHAVASSIEU, Assr. to SOC. POUR LA FABR. DE LA SOIE RHODIASETA (U.S.P. 1,622,368, 29.3.27. Appl., 26.3.26. Conv., 20.1.25. Cf. E.P. 246,430).—Aqueous solutions of inorganic and organic thiocyanates and thiocarbimides (*iso*thiocyanates) readily dissolve cellulose esters and ethers. The solutions so obtained are applied to the production of filaments of artificial silk by spinning into water in the usual way. T. S. WHEELER.

Tasteless, odourless, water- and fat-soluble printings on parchment paper. H. WREDE (G.P. 438,075, 19.12.25).—Solutions of cellulose or cellulose derivatives into which colouring matters, metal powders, etc. have been incorporated, are used for printing on parchment paper, the colouring matters etc. being such as are unattacked by the chemicals used in parchmentising. Alternatively, the incorporated substances may be such as give in the parchmentising bath insoluble or uncoloured, dyeing or non-dyeing, precipitates; or the dyes may be applied in colloidal form; or in place of cellulose and its derivatives, substances may be used which give parchment-like precipitates with the usual parchmentising agents; or the prints may be made before parchmentising. C. HOLLINS.

Production of wood pulp. W. D. MOUNT (E.P. 269,256, 13.10.25).—See F.P. 607,726; B., 1927, 185.

Drying apparatus (E.P. 269,064).—See I.

Wool oil emulsions (E.P. 246,867).—See XII.

Printing inks (E.P. 268,966).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Dyeing textile materials. H. B. SMITH (U.S.P. 1,623,709, 5.4.27. Appl., 29.8.23).—Cotton in the form of cloth or skeins with portions of varying degrees of hardness is immersed in a bath containing a restricted amount of a cotton dyestuff and an assistant of dyeing, *e.g.*, sodium chloride. The soft and loosely twisted portions of the cotton are rapidly dyed, and the bath is exhausted before the hard portions are coloured. The bath is then heated at 60° to fix the colour. The undyed parts of the material are bleached by immersion in an alkaline oxidising bath. A variegated material with good contrast between the dyed and undyed portions is obtained. T. S. WHEELER.

Dyeing acetate silk. BRITISH DYESTUFFS CORP., LTD., A. SHEPHERDSON, and A. DAVIDSON (E.P. 268,933, 1.3.26).—Acetate silk is dyed directly in presence or absence of dispersing agents by means of cœramidines, the anthraquinoneacridines obtained from α-arylaminoanthraquinones by the methods of G.P. 126,444 and 262,469. Examples are the cœramidines from 1-anilinoanthraquinone (greenish-yellow), 1-anilino-2-methylanthraquinone (golden-yellow), 1 : 5-dianilinoanthraquinone (pink), 1 : 4-di-*p*-toluidinoanthraquinone (brownish-red), 1-amino-4-*p*-toluidinoanthraquinone (bluish-red), 1-amino-5-*p*-toluidinoanthraquinone (violet). The 1-amino-5-*p*-toluidinoanthraquinone is prepared from 1-chloro-5-aminoanthraquinone and *p*-toluidine in presence of sodium acetate. C. HOLLINS.

Dyeing cellulose esters and ethers. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 245,790, 8.1.26. Conv., 9.1.25).—Cellulose esters or ethers are dyed with monoazo dyes made by coupling a diazo compound containing not more than one sulphonic group with *m*-aminoacetanilide or its homologues or derivatives which are capable of coupling. *m*-Aminoacetanilide is coupled with diazotised 2-chloroaniline-5-sulphonic acid (yellow) and with diazotised 3-nitroaniline-4-sulphonic acid (golden-yellow). C. HOLLINS.

Dyeing furs. BRITISH DYESTUFFS CORP. LTD., and R. S. HORSFALL (E.P. 268,952, 24.3.26).—Fur is dyed

from an acid bath with N-alkyl-ω-sulphonic acid derivatives of aminoanthraquinones, aminoazo and diaminoazo compounds, no other sulphonic group being present. Thus p-nitrobenzeneazophenylaminomethane-ω-sulphonic acid acid gives an orange-scarlet shade. The affinity of these dyes for fur is better than that of ordinary acid dyes, and they possess other advantages.
C. Hollins.

Dyeing animal fibres and fabrics of a protein nature. S. W. Wilkinson, Assr. to Zair Syndicate, Ltd. (U.S.P. 1,628,484, 10.5.27. Appl., 10.9.25. Conv., 11.9.24).—See E.P. 242,027; B., 1926, 11.

Liquor circulating means for treating materials with liquids (E.P. 269,241).—See I.

Dyeing of leather (U.S.P. 1,628,160).—See XV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Analysis of concentrated and fuming sulphuric acids by thermometric titration. T. Somiya (Proc. Imp. Acad., Tokyo, 1927, **3**, 76—78).—The water content of concentrated sulphuric acid may be determined by titration with fuming sulphuric acid containing 25% of free sulphur trioxide, the titration being carried out in a large test tube placed in a Dewar flask; the end-point is ascertained by plotting the temperature of the acid against the c.c. of fuming acid added and noting the point of intersection of the two straight lines thus obtained, the temperature ceasing to rise when all the water is converted into sulphuric acid. Fuming sulphuric acid is analysed in a similar manner; an excess of 80% sulphuric acid is added to combine with all the sulphur trioxide, and the mixture then titrated with the standard fuming acid. A. R. Powell.

Analysis of commercial acetic anhydride containing little or no sulphuric acid by thermometric titration. T. Somiya (Proc. Imp. Acad., Tokyo, 1927, **3**, 79—81).—Acetic anhydride, when free from sulphuric acid, is accurately determined by thermometric titration (cf. preceding abstract) with a 25% solution of aniline in acetic acid. The aniline solution may be standardised by thermometric titration with sulphuric acid or with a standard acetic anhydride. Alternatively, the acetic anhydride to be analysed is titrated with an unstandardised aniline solution, a second portion is partially hydrolysed with a known small quantity of water, and the remaining anhydride titrated against the aniline solution; if a is the volume of aniline used in the first titration for 1 g. of the anhydride, m the weight of anhydride used in the second test and, after hydrolysis by n grams of water, b the volume of aniline used in the second titration, then the percentage of acetic anhydride in the commercial sample is $10{,}210\, am/\{18{\cdot}02(an - b[n + m])\}$. If the sample contains sulphuric acid (present as sulphoacetic acid) the end-point is reached when the following reactions have taken place: (a) $Ph{\cdot}NH_2 + (CH_3{\cdot}CO)_2O = Ph{\cdot}NH{\cdot}CO{\cdot}CH_3 + CH_3{\cdot}CO_2H$, and (b) $Ph{\cdot}NH_2 + HSO_3{\cdot}CH_2{\cdot}CO_2H = Ph{\cdot}NH_2,HSO_3{\cdot}CH_2{\cdot}CO_2H$. In a second portion of the sample the sulphoacetic acid is removed by addition of lead acetate and, without filtration, the acetic anhydride is determined alone by titration with aniline. Alternatively, the sample is titrated directly with a solution of aniline hydrochloride in acetic acid, the sulphoacetic acid present not interfering in this case. The last-named acid may be determined directly in commercial acetic anhydride by thermometric titration with barium acetate, the following reaction taking place: $Ba(OAc)_2 + 2HSO_3{\cdot}CH_2{\cdot}CO_2H = Ba(SO_3{\cdot}CH_2{\cdot}CO_2H_2) + 2AcOH$. A. R. Powell.

Treatment of pollucite and preparation of cæsium chloride. A. Kastler (Bull. Soc. chim., 1927, [iv], **41**, 428—434).—The cæsium in American pollucite, a non-radioactive mineral containing only traces of potassium, rubidium, and calcium, is extracted as follows:—The mineral is treated with concentrated sulphuric acid and extracted with warm water. On cooling, the extract deposits crystalline cæsium alum, which is decomposed by excess of barium hydroxide. The solution of cæsium hydroxide thus obtained is treated with carbon dioxide, filtered, and the filtrate evaporated to dryness. The residue of cæsium carbonate is purified by extraction with alcohol (which dissolves only the cæsium salt) and dissolved in hydrochloric acid. The volatility of cæsium chloride first becomes apparent at 700°. S. K. Tweedy.

Acid-resistance of asbestos. A. Calmon (Gummi-Ztg., **41**, 1861—1865).—The acid-resistance of asbestos is determined by measuring the loss in weight of asbestos fibres, less than 1 cm. in length, after treatment with acid under specified conditions. Hornblende asbestos, *e.g.*, blue Cape or amosite asbestos, is more resistant to the attack of mineral acids, either cold or hot, than serpentine asbestos, *e.g.*, Canadian, Rhodesian, or Russian asbestos. Asbestos in general resists the attack of weak acids, *e.g.*, acetic acid, hornblende asbestos being unattacked even at the b.p. The effect of acid on the mechanical properties of the material is also described.
B. W. Clarke.

Properties of tungsten carbide, W_2C. K. Becker and R. Hölbling (Z. angew. chem., 1927, **40**, 512—513).—Evidence for the existence of the ditungsten carbide, W_2C, prepared by heating an intimate mixture of tungsten or tungsten carbide, WC, with carbon in stoicheiometric proportions at temperatures over 1600°, is obtained by examination of the X-ray diagram. The diagram shows only faint lines corresponding with free carbon, tungsten, or tungsten carbide. The compound is readily attacked by chlorine at 400° with formation of tungsten hexachloride and graphite, whereas tungsten and tungsten carbide of the same grain size are almost unattacked. The conclusion of Westgren and Phragmén (A., 1926, 1084) that the material is a solid solution of carbon in tungsten is therefore negatived.
L. M. Clark.

[Preparation of] pure oxides and salts of tungsten and molybdenum. E. K. Jenckes (Trans. Amer. Electrochem. Soc., 1927, **51**, 299—308).—Ammonium paratungstate and tungstic acid containing less than 0·05% of impurity are obtained commercially from wolframite or scheelite. Wolframite is decomposed with sodium hydroxide and calcium tungstate is precipitated from the solution by addition of calcium chloride. The

calcium salt is treated with hydrochloric acid, and the resulting tungstic acid is dissolved in ammonia solution. From this solution the pure ammonium salt is obtained. Pure tungstic oxide is obtained by dehydration of the acid. For the production of molybdic oxide, molybdenite is roasted, the sulphur content finally being lower than 0·5%. The roast is leached with ammonia solution, iron, copper, and calcium are then removed from solution, and ammonium molybdate is obtained by crystallising or seeding.

L. M. Clark.

Industrial absorption of nitrogen oxides in the synthetic nitric acid industry. C. Toniolo (Chim. et Ind., 1927, **17**, 546—553).—See B., 1927, 251.

Patents.

Production of persulphuric acid and its soluble salts from sulphuric acid by electrolysis. Oesterreichische Chem. Werke G.m.b.H. (E.P. 265,141, 27.10.26. Conv., 28.1.26).—Sulphuric acid containing about 500 g./litre of concentrated acid is electrolysed at 20°, a current concentration of 400 amp./litre being used. A yield of over 70% is obtained. The per-salts are made from the corresponding sulphates.

W. G. Carey.

Manufacture of hydrocyanic acid. O. Liebknecht, Assr. to Roessler & Hasslacher Chemical Co. (U.S.P. 1,626,648, 3.5.27. Appl., 24.6.24).—A mixture of carbon monoxide and ammonia is passed in contact with a catalyst composed of a porous non-acid charcoal, the temperature of the reaction being maintained at 400—800°. H. Royal-Dawson.

Concentration of ammonium nitrate solutions. Azogeno S.A. per la Fabr. Dell' Ammoniaca Sintetica e Prod. Derivati, and C. Toniolo (E.P. 247,229, 8.2.26. Conv., 7.2.25).—The latent heat of ammonium nitrate is utilised for concentrating solutions thereof by dissolving the dry salt in a concentrated solution of ammonium nitrate, heating at 85—125°, and spraying the product into dry air. W. G. Carey.

Manufacture of basic chromium salts [for tanning]. I. G. Farbenind. A.-G., Assees. of Farbwerke vorm. Meister, Lucius, & Brüning (E.P. 251,267, 15.4.26. Conv., 24.4.25).—Basic chromium salts which are easily soluble in water and tan rapidly are made by combining a basic chromium salt, or the hydroxide, with an oxide, hydroxide, or salt of an alkaline-earth metal or of zinc, magnesium, or aluminium, with the addition of acid or alkali to produce the desired basicity.

W. G. Carey.

Production of metal nitrides, hydrides, etc. É. Tilche (F.P. 611,139, 27.5.25).—In processes such as the production of iron nitride and subsequent conversion of it into iron and ammonia by treatment with hydrogen, the gases are subjected to the action of radioactive material or an emanation, and the iron is raised to a high electrical potential and heated to redness during treatment with the nitrogen. L. A. Coles.

Process and apparatus for preparation of carbon disulphide. H. Schulz (G.P. 438,037, 3.4.25. Addn. to G.P. 407,656; B., 1926, 823).—The prior process is modified (*a*) by feeding sulphur vapour into the upper part of the oven and withdrawing carbon disulphide from the lower part, or (*b*) by removing the carbon disulphide at slightly increased pressure, or (*c*) by adding the sulphur from two containers or tubes attached to the floor of the oven. C. Hollins.

Preparation of hydrogen sulphide. A. Henwood, Assr. to R. M. Garey (U.S.P. 1,623,942, 5.4.27. Appl., 9.11.25).—A mixture of sulphur (36%), paraffin b.p. above 110° (14%), and asbestos fibre (50%) evolves hydrogen sulphide in a regular stream when heated.

T. S. Wheeler.

Manufacture of stable hydrogen peroxide. A. L. Halvorsen (U.S.P. 1,627,325, 3.5.27. Appl., 2.7.21).—The peroxide of an alkali-forming metal is fed into a mixture of alcohol and sulphuric acid, forming hydrogen peroxide and a sulphate; the latter is collected on a filter, and the greater part of the solution is distilled in one operation, the vapours from which are condensed separately, forming a solution rich in hydrogen peroxide, and another rich in alcohol. H. Royal-Dawson.

Stable mixtures for generating carbon dioxide. J. F. Schwarzlose Söhne G.m.b.H., Assees. of Seydel (G.P. 437,113, 24.12.25).—Mixtures of carbonates with succinic anhydride, benzoic anhydride, cinnamic anhydride, tannin, etc. are used for the preparation of compressed medicaments, mouthwashes, etc.

S. S. Woolf.

Production of concentrated sulphur dioxide. Ges. für Linde's Eismaschinen A.-G. (G.P. 437,910, 14.3.25).—The temperature attained during the combustion of sulphur in oxygen is kept at a minimum by using an excess of sulphur vapour, and, if necessary, by carrying out the combustion under raised pressure.

L. A. Coles.

Dissolving liquid chlorine. G. Ornstein (G.P. 437,689, 12.4.25).—Liquid chlorine is added in the desired quantity to a flowing stream of a solvent, such as water, or an absorbent, such as an alkaline solution.

L. A. Coles.

Lime kilns (E.P. 265,654 and U.S.P. 1,627,215).—See IX.

Nitric oxide from air (E.P. 269,046).—See XI.

Fungicides (G.P. 436,923).—See XVI.

VIII.—GLASS; CERAMICS.

Immiscibility in silicate melts. J. W. Greig (Amer. J. Sci., 1927, [v], **13**, 1—44, 133—154).—Experiments in which binary mixtures of silica with various metallic oxides were heated at various high temperatures and the phases present after quenching determined by microscopical examination, show that silica-rich mixtures of magnesium, calcium, strontium, manganous, zinc, ferrous, nickelous, or cobaltous oxides with silica melt to two immiscible liquids, one of which is almost pure silica. The equilibrium relations in the affected parts of the systems: $MgO-SiO_2$, $CaO-SiO_2$, $SrO-SiO_2$, and $FeO-SiO_2$ have been investigated, and the equilibrium diagrams constructed. In all four cases the temperature of equilibrium between cristobalite and the two liquid phases is of the order 1690—1700°, and the silica ends of the equilibrium diagrams are markedly

similar. In each case cristobalite crystallises out from the less siliceous liquid so rapidly on cooling as to make the quenching to a clear glass very difficult. Experiments with barium oxide–silica mixtures show that these oxides are apparently completely miscible, although the shape of the liquidus of cristobalite is indicative of the proximity of an area of immiscibility. This type of liquidus has not hitherto been encountered in silicate studies. Beryllium, stannic, lead, aluminium, sodium, and potassium oxides are completely miscible. Investigations on the systems: $MgO-CaO-SiO_2$, $MgO-FeO-SiO_2$, $MgO-Al_2O_3-SiO_2$, $MgO-Na_2O-SiO_2$, $CaO-Al_2O_3-SiO_2$, $CaO-Na_2O-SiO_2$, and $MgO-K_2O-SiO_2$ show that in ternary systems containing two of the metallic oxides which form two immiscible liquids with silica, the range of compositions of mixtures which melt to form two liquids extends across the corner of the diagram, whilst for those ternary systems containing an oxide of the "miscible" type, there is only a very narrow range of compositions which melt to form two liquids. Cristobalite is the first crystalline phase to form when any of these liquids is cooled. Mixtures of boric and silicon oxides of varying composition were made into clear homogeneous glasses, there being no evidence of immiscibility. The quartz dissolved very slowly, and this is believed to be the reason why silica has been reported to be insoluble in fused boric oxide. This slow dissolution together with the settling of the heavier quartz through the melt probably explains the two layers reported by Dimbleby, Hodkin, Parkin, and Turner (B., 1923, 551 A). In so far as liquid miscibility with metallic oxides is concerned, silica is remarkably similar to boric oxide. All oxides found to be incompletely miscible with silica were found by Guertler (A., 1904, ii, 614) to be incompletely miscible with boric oxide. In each case, moreover, one of the liquids was found to be almost pure boric oxide. The m.p. of cristobalite is $1713 \pm 5°$. The equilibrium temperature between cristobalite and the two liquids in the system $CaO-SiO_2$, $1698 \pm 5°$, is suggested as a fixed thermometric point. The exact ratio of lime to silica is of little importance, provided that it lies within the range of formation of two liquids. A mixture 15 CaO, 85 SiO_2 is suitable. Purity of materials is, however, of the utmost importance. The application of the present data to the problems of silica refractories and their bearing on petrology are discussed.

J. S. Carter.

Influence of foreign matter on the thermal expansion and transformation of silica. J. F. L. Wood, H. S. Houldsworth, and J. W. Cobb (Trans. Ceram. Soc., 1926, **25**, 289—308).—Additions (generally 2%) of various substances (mostly having relatively low m.p., and including lime for comparison purposes) were made to a Welsh (Bwlchgwyn) quartzite, ground to pass a 100-mesh I.M.M. sieve. Test pieces made from the various mixtures were fired to cones 9, 12, and 14. Data concerning the reversible thermal expansion, density, porosity, and refractoriness of these test pieces were obtained. It was found that boric acid, potassium carbonate, potassium chloride, potassium chromate, biotite, sodium felspar, lithium chloride, and ferric chloride facilitate the conversion of the quartz, the effects of boric acid, potassium carbonate, biotite, and borocalcite being most pronounced in modifying the reversible thermal expansion. The true sp. gr. is not a safe criterion of the reversible thermal expansion of the different mixtures. Apparently the solution of the quartz in the matrix varies, some of the silica maintaining the expansion characteristics of a glass. Thus, the densities of mixtures made with 5% of boric acid and with 2% of lime are not widely different, but they have very pronounced differences in reversible thermal expansions. The refractoriness of the different mixtures is not appreciably inferior to that of the lime-bonded mixture. Some mixtures show an increase, others a decrease of porosity with increase of firing temperature.

A. T. Green.

Influence of iron oxide in promoting the inversion of silica. W. J. Rees (Trans. Ceram. Soc., 1926, **25**, 314—320).—Analyses and microsections of the dark, ferruginous patches on well-fired silica brick containing iron oxide were contrasted with those of a normal portion of the brick. The photomicrograph of the medium-brown portion shows the presence of a large proportion of tridymite. The crystals, besides being more numerous, are also larger than those observed in the normal brick. The dark-brown portion of the patches shows still greater development of tridymite. Although the presence of iron oxide in a silica brick batch favours quartz conversion and accelerates the rate of tridymite formation, there is no indication that the ferruginous patches possess appreciably lower refractoriness. The presence of 2—3·5% of iron oxide in silica bricks for use in the open-hearth process may be advantageous, as the increased tridymite content will result in greater volume stability and reduced spalling tendency. Iron oxide in silica bricks for coke ovens is deleterious.

A. T. Green.

Behaviour of felspar and flint with acids and bases. E. W. Scripture, jun. (J. Amer. Ceram. Soc., 1927, **10**, 238—242).—20% suspensions of felspar and of flint were made up in a series of tall test tubes, treated with varying quantities of sodium hydroxide and hydrochloric acid, and allowed to remain for 18 hrs. The absorption of the acid or alkali was determined either by the titration of a portion of the clear liquid or by comparison with standard indicators. Flocculation or deflocculation was measured by three observations, viz., the height of the clear liquid, the height of the sediment, and the turbidity as determined by the evaporation of a 20 c.c. portion withdrawn from a level of 16 cm. below the surface (the total height of the column being 26 cm.). With large additions of alkali, flocculation, analogous to the so-called "salting-out" effect noted with clay suspensions, occurs. With smaller additions of alkali and passing over to the side of small acid additions, a wide deflocculation zone with constant p_H of suspension is obtained. This state is characterised by compact sediment and a turbid liquid above. More acid additions result in pronounced flocculation, the sediment being loose and the liquid clear, corresponding with the ordinary flocculated condition of clay suspensions. Within the usual range of p_H, clay causes a decrease, whilst the flint and felspar cause an increase of alkalinity, the effect being greater with felspar.

A. T. Green.

Variation of soda and boric oxide in ground-coat enamels. R. R. FUSSELBAUGH (J. Amer. Ceram. Soc., 1927, **10**, 270—274).—On the basis of a ground-coat enamel already in use three others were evolved, (*a*) with 5% of soda and 25% of boric oxide; (*b*) with 15% of both soda and boric oxide; and (*c*) with 25% of soda and 5% of boric oxide. The batches were carefully milled and aged, and other precautions taken to get identical circumstances. Three thicknesses of enamel were tested, and these were fired at 870° for $4\frac{1}{2}$, 3, and $2\frac{1}{2}$ min. respectively. The results show that there are definite limits to soda and boric oxide contents which can be used in this type of ground coat. An increase of soda at the expense of boric oxide produces an enamel liable to form "copperheads," but reduces the tendency to "blister" and "fish-scale." The converse happens when boric oxide is increased at the expense of soda. The phenomenon of blistering is discussed. A. T. GREEN.

Agreement of ground coat and enamel. A. MALINOVSZKY (J. Amer. Ceram. Soc., 1927, **10**, 275—277).—Staley's ratio is considered an admirable device for the control of enamelling. To obtain this factor, a "refractory value" of the ground coat, which is the ratio of the percentage of refractory constituents (in the ultimate analysis) in the ground coat to the percentage of fluxing constituents, is first determined. A "fluxing value" of the enamel, which is the inverse of the refractory value, is next obtained. Staley's ratio is found by dividing the refractory value of the ground coat by the fluxing value of the enamel. Good control has been obtained by keeping this factor at a constant value ranging between 1·33 and 1·66. A. T. GREEN.

Influence of iron oxide and iron sulphides on the rate of quartz conversion in commercial silica brick manufacture. W. HUGILL and W. J. REES (Trans. Ceram. Soc., 1926, **25**, 309—313).—Silica bricks were made on a commercial scale from (*a*) marcasite-incrusted ganister and (*b*) normal ganister free from iron sulphides, with lime-emulsion additions to give 1·5% CaO in the finished product. These bricks were fired to cone 15—16. The results show that iron sulphides help the quartz conversion considerably, and appear to increase the mechanical strength of the product. Iron oxide was removed from a portion of a batch of ganister by means of treatment with acids, and bricks were made of the treated and untreated ganisters. It was thus shown that the presence of iron oxide in the brick favourably influences the rate of quartz conversion and increases the mechanical strength of the resultant product. A. T. GREEN.

Hydrogen-ion control in ceramics. W. A. TAYLOR (J. Amer. Ceram. Soc., 1927, **10**, 243—258).—The meaning of the p_H of solution and the factors influencing it are demonstrated, and a description of the indicator method of its evaluation is given. A. T. GREEN.

Testing refractory materials for resistance to slag corrosion and erosion. A. J. DALE (Trans. Ceram. Soc., 1926, **25**, 326—338).—Many testing methods concerning slagging have been evolved, but there is little fundamental data. A discussion of the testing methods is given. These are classified, and include the following measurements: (1) depth of penetration of slag into brick; (2) viscosity of the slag brick solution; (3) volume of brick dissolved or otherwise removed during a definite heat-treatment in contact with slag; (4) effect of impact of powdered and heated slag on heated refractory material; (5) change in ultimate chemical composition of the slag or of the brick, after a definite period of reciprocal interaction; (6) progressive alteration in temperature gradient through a wall, one face of which is exposed to the solvent action of the corrosive melt; and (7) a deductive method based on the micro-examination of the layer between the slag and brick. It is suggested that laboratory methods which would evaluate the progressive rate of corrosion and erosion of different refractories by slags at various temperatures should provide results of definite industrial value. A. T. GREEN.

Thermal properties of refractories, and factors influencing them. A. T. GREEN (Trans. Ceram. Soc., 1926, **25**, 361—385).—A *résumé* of the data concerning the sp. heat, heat capacity, temperature diffusivity, thermal conductivity, and surface emissivity of refractories is given. Some of the factors governing these properties are discussed. The average surface emissivity for a firebrick surface between 200° and 500° is given as 0·72. A. T. GREEN.

Trend of design in modern coke-oven construction and its bearing upon refractory materials. A. E. J. VICKERS and A. T. GREEN (Trans. Ceram. Soc., 1926, **25**, 407—427).—The principles involved in modern coke-oven design, chiefly including the supply, utilisation, and transmission of heat, the velocity of the gases in the heating flues, the recovery of heat, and certain structural features, are discussed. A *résumé* of the properties of silica bricks, with special reference to coke-ovens, is given. Silica bricks are shown to possess certain advantages over fireclay and siliceous refractories, particularly those relative to mechanical strength at high temperatures, temperature diffusivity, thermal conductivity at high temperatures, and resistance to corrosion. A. T. GREEN.

Spalling of refractory materials. A. T. GREEN and A. J. DALE (Trans. Ceram. Soc., 1926, **25**, 428—470).—Spalling is defined as the inability of a refractory material to withstand without disrupture the stresses induced by temperature fluctuations. A fundamental equation shows the spalling tendency to be directly proportional to the coefficient of expansion and inversely to the maximum shearing strain and the square root of the coefficient of temperature diffusivity. The constitutional factors influencing the spalling of silica, siliceous fireclay, magnesite, chromite, sillimanite, and zirconia products are discussed. The relation between the thermal expansion and the constitution of refractories, particularly silica and fireclay products, is considered at length. The methods which have been formulated for the testing of spalling are reviewed. It is stated that the crux of the solution of the problem lies in the study of the elasticity, visco-elasticity, and plasticity of different refractories over a complete range of temperature. The influence of iron oxide in promoting what is apparently a type of spalling encountered in the carbonising industries receives attention. A. T. GREEN.

Abstraction of sulphur dioxide from flame gases by glasses and glazes. Faber.—See II.

Patents.

Heat-resisting glass. R. F. Brenner, Assr. to H. C. Fry Glass Co. (U.S.P. 1,623,301, 5.4.27. Appl., 9.5.22).—A heat-resisting glass contains silica 65—80%, boron trioxide 10—20%, colloidal aluminium oxide 2—10%, alkaline oxides 5—10%, and alkaline-earth oxides 0·5—2%. T. S. Wheeler.

Blowing and moulding of silica articles. Quartz & Silice (E.P. 263,766, 22.11.26. Conv., 29.12.25).—An electrically heated furnace for softening silica is provided with a balance arm supporting the silica blank and a counterweight, the movement of which indicates the deformation and therefore the softening point of the blank. Below the furnace is situated a mould, to which the softened blank is rapidly transferred, and in which it is blown to the required shape by means of compressed air. No subsequent machining or superficial fusion is necessary to produce a smooth finish. B. W. Clarke.

Manufacture of clay-bonded products by means of a casting clay slip. Scheidhauer & Giessing A.-G. (E.P. 262,383, 24.6.26. Conv., 4.12.25. Addn. to E.P. 253,947; B., 1926, 254).—In the manufacture of clay-bonded products according to the main patent, other non-plastic refractory substances in a fine state of division, such as sillimanite, calcined alumina, magnesite, etc., which are suitable for clay-bonding, are substituted for the chamotte and mixed with a small proportion of bonding clay for use as a slip during the casting process. B. W. Clarke.

Manufacture of decorated tiles. V. Lefebure (E.P. 268,851, 7.12.25).—A mixture of an alkaline silicate, colloidal silicic acid, or silica itself with a metallic oxide or salt, one of the ingredients being in a syrupy condition, is moulded to a sheet or applied to an asbestos cement or other backing. The sheet thus formed is heated at below 350° to harden the silicate layer. Pigments and other fillers may be incorporated if required. B. W. Clarke.

Making and moulding hard porcelain. A. M. McIntosh (U.S.P. 1,627,245, 3.5.27. Appl., 15.7.22).—The finished porcelain is heated until it is plastic, then pressed and moulded. H. Royal-Dawson.

Preparation of ceramic products. W. Vershofen (U.S.P. 1,628,522, 10.5.27. Appl., 26.9.23. Conv., 27.9.22).—See G. P. 380,623; B., 1924, 295.

Treatment of metallic oxides (E.P. 248,360).—See X.

IX.—BUILDING MATERIALS.

Evaporation of water and salt solutions from surfaces of stone, brick, and mortar. A. P. Laurie and J. Milne (Proc. Roy. Soc. Edinburgh, 1926, 47, 52—68).—Experiments were made to test the hypothesis that if in a porous substance (whether stone, brick, mortar, or brick or stone with mortar) containing an equal amount of water distributed through it the evaporation from one part of the surface is slower per unit surface than from the other, then there will be a passage of water from the slow-drying to the quick-drying portion during the drying-out. This was confirmed in the case of a porous sandstone, and it was also shown that in the case of untreated stone and stone treated with "silicon ester" two stages of evaporation are apparent. In the first stage, when the stone is wet and the surface is probably kept flooded by capillary attraction, stones of equal area evaporate at approximately the same rate, the rate for each stone being roughly proportional to the amount of water left. In the second stage there is a much faster removal of residual water from the untreated stone than from the treated stone. In the case of brick in contact with mortar, decay of the brick is due only to crystallisation below the brick surface of calcium sulphate, the sulphuric acid being derived from the air and rain. The principal source of lime is in the mortar, the acid-charged rain passing into the mortar and through the brick in contact with the mortar, and forming calcium sulphate. During evaporation the solution of calcium sulphate is drawn into the brick and crystallises there, owing to the rate of evaporation at the brick surface being faster than the rate at the mortar surface. Study of a portion of an old priory which had been pointed with a mixture of lime and Portland cement led to the following conclusions: In pointing old buildings consisting of sandstones containing calcite, care must be taken to use a sand free from salt. Portland cement is quite unsuitable for such a purpose as it makes a dense mortar with a very low rate of evaporation. The practice of washing the surface of the mortar and not leaving it with the skin formed by the trowel is probably wise, as the skin probably diminishes the rate of evaporation. Before re-pointing a building, after the joints have been cleaned out, a suitable preservative should be sprayed on the surface of the brick or stone. It is essential that both mortar and stone should not be treated with a preservative, as this reduces the rate of evaporation for both surfaces. Experiments throwing light on the conditions promoting local crystallisation below the surface are described. W. Clark.

Painting cement and plaster. Gardner.—See XIII.

Patents.

Process and apparatus for burning cement. J. S. Fasting (E.P. 268,868, 6.1.26).—A rotary kiln is provided with a series of tubes or channels arranged on the outside of the drying zone kiln tube and which communicate freely with the body of the kiln. The raw material to be dried and the combustion gases pass in opposite directions through these tubes, which, owing to their increased cross-sectional area, will reduce the velocity of flow of the combustion gases and give more efficient heat transference. The material after being dried is subjected to a grinding process by passing below a device located in the tubes, thereby facilitating the subsequent burning process. B. W. Clarke.

Kilns [for burning limestone]. E. W. Pattison. From W. D. Mount (E.P. 265,654, 12.8.25).—A vertical kiln comprises the following parts in order downwards: Charging hopper with bell, magazine for limestone, outlets for gases, upper part of kiln with peep holes, constricted part of kiln with downwardly-inclined gas tuyères just below the narrowest part, lower part of

kiln, rotating cone bottom discharging burnt lime. Air for combustion enters through the burnt lime, and is thus preheated; the gas inlets are inclined downwards in order to make the flame reach the centre and prevent overburning up the sides. B. M. Venables.

Lime kiln and method of burning lime. A. E. Truesdell, Assr. to Doherty Research Co. (U.S.P. 1,627,215, 3.5.27. Appl., 23.4.23).—The kiln is connected with the vertical kiln of a furnace for burning fuel under such conditions that the products of combustion are obtained at a temperature in excess of that required for burning limestone, and delivered to the kiln. The waste gases leaving the kiln are re-introduced into the furnace between the point where combustion takes place and that of delivery of the products of combustion to the kiln. H. Royal-Dawson.

Concrete and similar materials. K. P. Billner (E.P. 268,893, 12.1.26).—Cement is mixed with from 70 to 100% of water and from 0·03 to 0·5% of powdered aluminium or zinc together with fine aggregate and colouring materials if required, sufficient water being added so that the concrete can be applied by brushing or spraying to brickwork, ordinary concrete, and structural steelwork. B. W. Clarke.

Manufacture of hydraulic cement and the like. O. Gerlach (U.S.P. 1,627,170, 3.5.27. Appl., 9.10.24).—A mixture of calcium carbonate, zinc retort ashes, and carbon is calcined at 950—1000°. Zinc is volatilised and burns to oxide which is deposited on the lime, the product being finally pulverised. F. G. Crosse.

Artificial stone or marble. L. E. Welch (U.S.P. 1,626,577, 26.4.27. Appl., 17.8.26).—Magnesium oxide and a filler are mixed with a suitable proportion of a liquid consisting of 1⅝ gals. of zinc sulphate solution (*d* 2·1) and 100 gals. of magnesium chloride solution (*d* 1·8—2·4) together with acetic acid. B. W. Clarke.

[Granulation] treatment of slags by fluids under pressure. M. Maguet, Assr. to Soc. Anon. des Chaux et Ciments de Lafarge et du Teil (U.S.P. 1,627,982, 10.5.27. Appl., 6.10.23. Conv., 26.10.22).—See E.P. 206,132; B., 1925, 176.

[Mould for] the production of artificial stone blocks. H. Schaefer (E.P. 268,587, 7.6.26).

Roofing material. Flintkote Co., Assees. of C. E. Rahr and R. E. Drake (E.P. 255,477, 16.7.26. Conv., 16.7.25).

Cement for electrodes (G.P. 437,552).—See XI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Alloys of iron research. W. Rosenhain. **V. Preparation of pure chromium.** F. Adcock. **VI. Preparation of pure manganese.** (Miss) M. L. V. Gayler. **VII. Preparation of high-purity silicon.** N. P. Tucker (Iron and Steel Inst., May, 1927. Advance copy. 52 pp. Cf. B., 1924, 832; 1925, 829).—V. Methods for the preparation of very pure chromium are described. The starting point is electrolytic chromium made by the electrolysis of an aqueous solution containing 30% of pure chromic acid and 1% of sulphuric acid. Commercial "pure" chromic acid is unsuitable, as it contains sulphate. Full details of the electrolysis are given. Three processes have been developed, all using lead anodes in connexion with (i) a porous cell and tin cathode, (ii) a square steel bar cathode, (iii) a rotating steel tube cathode. Of these, (i) gives a metal of high purity in dendritic form, unsuitable for melting in the induction furnace, whilst excessive electrical energy is used. Method (ii) is the simplest for obtaining small quantities of pure metal in a compact form, and the electrical energy used is only about one quarter that required by the first process. It is, however, difficult to remove the metal from the cathode. Method (iii) is the best for production on a larger scale, and gives an easily-removed compact deposit with moderate energy consumption. The electrolytic metal contains both hydrogen and oxygen, the latter being present in a form which leaves no residue on dissolution in acid. The hydrogen is removed by melting *in vacuo*, but this process changes the state of the oxygen to insoluble chromic oxide, which can only be removed by heating the solid metal in a current of pure dry hydrogen at 1500—1600°, when a product is obtained which is spectroscopically pure. It cannot, however, be melted, even in fused alumina crucibles *in vacuo*, without introducing oxygen. Pure chromium is quite soft, and its m.p. considerably above that of iron.

VI. Manganese of 99·3% purity may be prepared by the reduction of trimanganese tetroxide with aluminium in a salamander crucible lined with alumina; manganous oxide is not so suitable for this purpose. The method of Allmand and Campbell (A., 1925, ii, 305) is also suitable. Manganese with total impurities less than 0·01% can, however, be readily prepared by distilling commercial manganese in a high-frequency induction furnace under a pressure of 1—2 mm. The resulting metal, silver-grey in colour, is hard enough to scratch glass, but very brittle. Its m.p. is 1244° ± 3°. It shows four transformation points at the following temperatures:—(1) 1191° ± 3°; (2) 1024° ± 2°; (3) 742° ± 1°; and (4) 682° ± 1°. Of these changes, (3) is accompanied by a change of crystal structure, but not (4). One or both of these is accompanied by a marked change in volume. The ingots obtained by melting *in vacuo* are extensively cracked owing to the transformations on cooling. The impure thermit manganese is less brittle, and has only a few cracks, whilst its m.p. and transformations are at higher temperatures than in the pure metal.

VII. The greater part of the impurities in commercial silicon lie in the grain boundaries, and may be removed by the following process. The best quality of commercial silicon is finely powdered and just covered with water. Hydrochloric acid is cautiously added, when the silicide impurities are decomposed with evolution of spontaneously inflammable gases. The product is digested with hot aqua regia for 24 hrs., and then washed and transferred to a platinum dish, where it is treated with sulphuric and hydrofluoric acids, and heated until it fumes. After a further treatment with concentrated hydrochloric acid, it is washed and dried. The final product under favourable circumstances

contains 99·94% Si, and may be melted in silica crucibles. W. Hume-Rothery.

Alloys of iron research. VIII. Constitution of alloys of iron and phosphorus. J. L. Haughton (Iron and Steel Inst., May, 1927. Advance copy. 18 pp.). —The constitution of alloys of iron and phosphorus has been determined by thermal and microscopic analysis in the range 0—30% P. The addition of phosphorus lowers the f.p. of iron, which falls to a eutectic point at 1050°, and contains, approximately, 10·5% P; the constituents of this eutectic are the α solid solution of phosphorus in iron, and the compound Fe_3P. The liquidus then rises to 1166°, at which temperature the compound Fe_3P is formed by a peritectic reaction between the liquid and the compound Fe_2P, the latter corresponding to a maximum on the liquidus at 1380°. Beyond this point the liquidus falls to a second eutectic, the constituents of which are Fe_2P and an unknown compound. The compound Fe_3P forms no solid solutions, but magnetic analysis shows that it undergoes a transformation at about 420°, which is similar to the corresponding transformation in cementite (Fe_3C). The compound Fe_2P forms no solid solutions with excess of iron, but there may be a very slight range of solid solubility on the phosphorus side. The effect of phosphorus on the critical points is very marked. The A4 point is rapidly depressed and the A3 point raised, the two joining to form a completely enclosed area representing the existence of γ-iron. There is thus no boundary between α- and δ-iron, both of which form the same space lattice. W. Hume-Rothery.

Temper-hardening in steels. T. Matsushita and K. Nagasawa (Iron and Steel Inst., May, 1927. Advance copy. 16 pp.).—A plain carbon steel containing 0·75% C was quenched from 800°, and its magnetic hardness, *Hm* (measured by the coercive force in gauss), was determined after tempering for different times at various temperatures. When tempered below 400°, *Hm* decreases with lapse of time, reaching an asymptotic value. But between 400° and 500° the *Hm*–time curves first decrease, then rise to a maximum, and finally gradually diminish. The *Hm*–temperature curves fall as the temperature is raised to about 400°, rise to a maximum of *Hm* between 450° and 500°, and then fall again. The results with nickel–chromium steels are similar with different temperature values. Corresponding abnormal changes within the same temperature ranges have been found for the impact hardness, Brinell hardness, and tensile properties. The annealing of cold-worked steels produces similar abnormal changes in the same temperature zones. The cause is probably "recrystallisation," causing the disintegration or refining of the iron crystals before crystal growth begins. The magnetic hardness and mechanical hardness curves run parallel to each other. W. Hume-Rothery.

Fatigue strength of hard steels. J. M. Lessells (Trans. Amer. Soc. Steel Treat., 1927, **11**, 413—424).—Tests on 0·42% and 1% carbon steels quenched and slightly tempered to give a ball hardness over 400 show that when a departure occurs from a straight line relation between tensile strength and Brinell hardness the ratio of the endurance limit to the tensile strength is also low. This effect was associated with residual compressive stresses on the outside of the specimens. T. H. Burnham.

Drawing of steel wire and its relation to qualities of steel. E. A. Atkins (Iron and Steel Inst., May, 1927. Advance copy. 24 pp.).—The action of drawing wire causes a relatively quicker flow of metal in the interior than at the outside of the wire, and consequently any steel having segregations at the core of the rods leads either to complete or incipient fractures. Surface defects on wire may be caused by surface splashes or skin blow-holes on ingots, or by faulty after-treatment, *e.g.*, production of fins or cracks during rolling. One serious source of wire "running out" or not sizing correctly has been traced to the presence of crystalline alumina in steels which have been deoxidised with aluminium. Thus one mild steel which exhibited about 40,000 specks to the sq. in. increased in diameter 10 mils during the last pass when being drawn to 14 gauge. Deterioration of wire may be caused by the action of occluded hydrogen on inclusions during the pickling process, as also on the deposited metal after galvanising, tinning, or electro-plating. Trouble has been caused occasionally through the presence of abnormal amounts of copper, tin, or nickel in wire-making steel. C. A. King.

Theory of the growth of cast iron repeatedly heated. C. Benedicks and H. Löfquist (Iron and Steel Inst., May, 1927. Advance copy. 31 pp.).—Increase in volume is necessarily brought about by a difference in expansion between adjacent layers in a metal due to sudden temperature changes. No growth will occur unless cast iron is subjected to repeated heat cycles, including the critical temperature, and on account of internal bursting around the graphitic lamellæ the anomalous contraction at Ac decreases more rapidly than the anomalous expansion at Ar, thereby giving rise to a residual expansion with every heating cycle. Successively increasing internal fracturing in the metal permits the access of oxidising gases, which occasions further growth, as does also the increase in volume due to the formation of temper carbon. Growth of cast iron on heating may be reduced by stabilising cementite in white iron, as, *e.g.*, low carbon and silicon and high manganese content, or by the introduction of 3—4% Cr. In grey iron conditions and composition which tend either to increase the tensile strength or raise the transformation point should be favoured. C. A. King.

Heat-resisting steels. W. H. Hatfield (Iron and Steel Inst., May, 1927. Advance copy. 26 pp.).—The corrosion-resistance at high temperatures of a number of pure metals and ferrous alloys was investigated by measuring their increase in weight from exposure at 900° in air with various gaseous additions, and in industrial gases. A complex furnace gas was more active than a muffle atmosphere. Water vapour and sulphur dioxide in the gases increased the attack. The order of resistance of the pure metals was chromium, nickel, cobalt, copper, iron, tungsten. The protective influence of chromium on iron was greater than that of nickel, but the addition of 8% of nickel to 18% chromium steel improved the resistance of the latter,

the optimum results being obtained by the further addition of 4% of silicon or tungsten. Silicon–chromium steel was strongly resistant in all the gases used. Nichrome, which was added for comparison, failed badly in sulphur dioxide, in which, however, 15% silicon iron was substantially resistant. T. H. Burnham.

Cold-rolled strip steel. T. Swinden and G. R. Bolsover (Iron and Steel Inst., May, 1927. Advance copy. 29 pp.).—Tests on the cold-rolling of strip steel are recorded. The steels contained 0·10, 0·34, 0·54 and 0·70% C, and were rolled from 0·30 in. to 0·10 in. in thickness. It was found that the maximum stress in tension increased with cold-working, but, for a given percentage reduction by cold-work, the increase was proportionately less as the amount of cold-work previously received by the steel increased. The proof stress rapidly approached the maximum stress and then followed more or less parallel to it; it became proportionately less as the carbon content increased. The resistance to shear increased with cold-working, but to a less degree than the maximum stress in tension. The percentage elongation value fell rapidly with initial cold-work, subsequent cold-rolling having little influence after an elongation of 10% had been reached. Brinell hardness tests on cold-worked materials are unreliable as an indication of the maximum stress in tension, but, in general, the ratio between these two tests increased with the cold-work. In general, the variation in properties was similar in the various steels, but the dead-soft steel (such as is used for cold-press work) was the least hardened by cold-work, whilst a 0·70% carbon steel, annealed before cold-rolling, work-hardened more readily. There was no indication of a fall in hardness with progressive cold-rolling. Five of these steels after reductions in thickness of approximately 33% and 66% were heated for 1 hr. at various temperatures up to 700° and the tests repeated. It was found that, except in the case of the dead-soft specimen, the proof stress rose on reheating to 300°, and the ratio of this to the maximum stress also increased; above this temperature both values fell. The rise in proof stress increased with the carbon content and with the amount of cold-work received. The maximum stress in tension increased with the reheating temperature up to 300° in all steels except the dead-soft strip, and fell with increasing temperature above this point. Annealing before cold-rolling reduced the rise obtained by reheating after rolling. The resistance to shear also increased up to 300°, the dead-soft strip being less affected than the remaining steels; the rise was again proportional to the amount of cold-rolling. The Brinell hardness rose on reheating, and the ratio to the maximum stress fell, thus giving a higher proportional rise for the Brinell number than for the maximum stress. The time of reheating beyond 1 hr. at 700° had a decided influence on the mechanical properties. Micrographs showing the change of structure in the steels are given.

M. E. Nottage.

Influence of annealing temperature on the properties of mild steel sheets. C. A. Edwards and J. C. Jones (Iron and Steel Inst., May, 1927. Advance copy. 36 pp.).—The effects upon the properties of steel sheets of the temperature range 750—920° reached during "black-annealing" were investigated. The Erichsen values of several samples, varying in thickness and composition, after annealing at temperatures between 400° and 1000°, have also been determined. It is shown that most of the stresses left in the sheets from the hot-rolling mills were removed by annealing at 625—650°. A further improvement generally occurred after the temperature was raised above the A3 point. In some cases a slight deterioration of the values was obtained by annealing at 700—875°, due to growth of the ferrite crystals. It is suggested that for those sheets in which the ferrite completely recrystallises at the low temperature, it may be possible so to adjust the period of annealing at 700° that small equiaxed crystals are obtained which will give results even better than those obtained by annealing above the A3 point. Tensile tests were carried out on sheets 0·4 mm. and 1·25 mm. thick, the tests being made both parallel and at right angles to the direction of rolling. It was found that after annealing below 550°, the yield-points across the grain were the higher. Annealing at 650° produced a fall in the yield-point, and the values across the grain became the lower. Above 700° the yield-point values decreased, possibly owing to the rapid growth of the ferrite crystals. At 850—900° a second rise in the value of the yield-point set in, which reached a maximum at the A3 point. The maximum stresses showed variations similar to the yield-points. The tensile strength measured at right angles to the direction of sheet-rolling was always the higher after annealing at the same temperature. There was a marked improvement in elongation after annealing at 600°, which reached a maximum at 650—700°; depreciation then set in, and a minimum was obtained at 800°. Annealing at still higher temperatures brought about secondary improvements, a maximum being reached at 900°; above this temperature there was a steady decrease in the elongations. At about 700° the elongations of the 0·4 mm. specimens were considerably higher when tested in the direction of the sheet-rolling, but the differences in this respect were small after annealing at 900°. The possibility of adjusting the ratio of the degree of bar-rolling to that of sheet-rolling applied to the material in directions at right angles to one another, so that the properties of the finished sheets should be equal in both directions after annealing at 700°, is suggested. The tensile properties of the 1·25 mm. specimens were more regular at right angles to the direction of the sheet-rolling, and the elongation values were also better. M. E. Nottage.

Alloys of iron and manganese containing low carbon. R. Hadfield (Iron and Steel Inst., May, 1927. Advance copy. 65 pp.).—A series of thirteen alloys with manganese content ranging from 0·06 to 38·9% were prepared, in which the carbon averaged 0·08%, together with two alloys containing 61·5% and 83·5% Mn. Tensile, bending, hardness, impact, and hammer-hardening tests were made on the alloys in three conditions, as cast or forged, annealed, and water-quenched. The electrical resistance, heating and cooling curves, and corrodibility of the alloys were determined, and photomicrographs of the series are given. The

alloys are divided into four groups:—0—4% Mn, soft and tough; 4—10% Mn, hard and brittle; 10—15% Mn, decreasing in hardness; and 15—39% Mn, possessing to a limited extent the characteristics of manganese steel. Manganese progressively reduces the magnetisability of the alloys, which between 16 and 17% become non-magnetic. Contrary to manganese steel, the specific magnetism is not increased by annealing at 500° for 60 hrs. The electrical resistance increases progressively with the manganese content. The loss of magnetism on heating is little influenced by the manganese percentage, but on cooling the intensity of the transformation diminishes, vanishing at 16—17% Mn before ordinary temperatures are reached. Between 1·68% and 38·9% Mn the microstructure of the forged alloys changes progressively—pearlitic, troosto-martensitic, martensitic, martensitic and austenitic, austenitic. No alloy possesses useful corrosion-resisting properties, and up to the present none has shown valuable properties for industrial application. T. H. Burnham.

Determination of manganese in cobalt steels. A. Hallbauer and P. Krüger (Z. angew. Chem., 1927, **40**, 513—514).—The presence of cobalt interferes with the direct determination of manganese in an alloy steel, and separation of manganese as dioxide by treatment of a solution of the steel with the usual oxidants is not completely satisfactory. More consistent results are obtained by precipitation of iron, chromium, tungsten, etc. from a nitric acid solution of the steel with zinc oxide. Cobalt is removed from the filtrate by precipitation as potassium cobaltinitrite. Manganese can then be determined by the usual method after removal of nitrous acid with carbamide. L. M. Clark.

Gallium in flue-dust. H. Ramage (Nature, 1927, **119**, 783).—Notable quantities of gallium have been detected in flue-dusts. The following elements, derived from South Yorkshire coal, have been detected in a gas-works flue-dust: lithium, sodium, potassium, rubidium, cæsium, copper, silver, calcium, strontium, zinc, aluminium, gallium, indium, thallium, carbon, titanium, silicon, lead, vanadium, phosphorus, arsenic, antimony, bismuth, oxygen, chromium, molybdenum, sulphur, manganese, chlorine, iron, and nickel. A. A. Eldridge.

Intercrystalline corrosion of metals. H. S. Rawdon (Ind. Eng. Chem., 1927, **19**, 613—619).—Intercrystalline corrosive attack of metals when due to corrosion alone can usually be related to some structural features peculiar to the metal. Thus pure tin shows little tendency to become brittle when corroded, but if traces of aluminium are present it becomes embrittled upon very slight corrosion, the attack being confined to the micro-constituent containing aluminium. A relationship of this kind has not been established in the case of the severe intercrystalline corrosion occurring with commercially pure lead. In general, metals become embrittled at a quicker rate when under high stress, and any practical remedy seems to include the reduction of the stress acting upon the metal, either externally or internally, considerably below the yield-point of the metal, and the application of protective coatings to prevent corrosion as far as possible. The work of other observers relating to the embrittlement of tin, lead, copper, brass, and iron is reviewed. C. A. King.

Mechanism of the production of zinc. M. Bodenstein (Trans. Amer. Electrochem. Soc., 1927, **51**, 449—456).—The reduction of zinc oxide to zinc in presence of carbon is shown to proceed according to the mechanism (i) $ZnO + CO = Zn + CO_2$, and (ii) $CO_2 + C = 2CO$. The velocity of the reaction is determined by the comparatively slow rate of the second stage, and thus depends largely on the physical condition of the charcoal, coke, or graphite which is acting as reducing agent. L. M. Clark.

Electro-deposition of tin. W. Fraine (Metal Ind. [New York], 1926, 24, 463; Chem. Zentr., 1927, I, 647).—For obtaining thick, smooth deposits of tin, the following electrolytic baths are recommended: (*a*) a solution of stannous hydroxide in fluoboric or fluosilicic acid; (*b*) a solution of 113 g. of stannous chloride crystals, 283 g. of sodium hydroxide, and 226 g. of dextrose in 4·5 litres of water; and (*c*) a solution of 680 g. of sodium stannate, 7 g. of stannous chloride crystals, and 0·1 g. of resin in 4·5 litres of water. A. R. Powell.

Principles of electrolytic studies on corrosion. W. Blum and H. S. Rawdon (Trans. Amer. Electrochem. Soc., 1927, **51**, 46—490).—From a discussion of the principles of corrosion it is shown that, if the corrosion is essentially electrolytic in character, it may reasonably be accelerated by the application of an *E.M.F.*, and that, in this case, the potential required to produce a certain current density or loss in weight is a much more accurate measure of the relative corrodibility of various metals than is the loss in weight produced by a given current in a given period. In the latter case, the relative losses in weight of different metals do not even approximately correspond with those obtained by simple immersion in corroding media. For studying electrolytic corrosion, it is suggested that two plates of the metal should form two opposite ends of a hard rubber box, and that the loss in weight of a third plate of the same metal immersed midway between the other plates should be measured when different *E.M.F.* are applied between the centre and outside plates. The results may be plotted as loss in weight–potential curves or as current density–potential curves. Examples of the use of the method are given. A. R. Powell.

Embrittlement of boiler plate. S. W. Parr and F. G. Straub (Ind. Eng. Chem., 1927, **19**, 620—622).—See B., 1926, 1016.

Influence of iron oxide on silica [bricks]. Rees.—See VIII.

Corrosion of metals by waters. Lührig.—See XXIII.

Patents.

Production of steel. C. H. Wills, Assee. of J. K. Smith (E.P. 245,716—7, 5.5.25. Conv., 10.1.25).—(a) Iron produced from ore according to E.P. 215,400 (B., 1924, 601) is separated from accompanying dross and the metallic particles are pressed into briquettes which form the charge for a crucible process. (b) Pig iron may be added to the briquette charge in the proportion of 15—25%. C. A. King.

Production of iron and other carbon-binding metals and alloys thereof having a very low percentage of carbon. H. G. E. CORNELIUS (E.P. 252,017, 10.5.26. Conv. 11.5.25).—Iron containing less than 0·05% C is produced by reducing a mixture of ore and carbonaceous material in briquette form in an electric furnace. The quantity of reducing material must be at least 5% less than the theoretical amount required. Suitable materials for regulating the viscosity and conductivity of the slag may be incorporated in the briquettes, and briquettes containing the ore of desired alloying elements may be introduced. C. A. KING.

Manufacture of steel and treatment of same for the manufacture of edge tools. A. E. G. T. VON VEGESACK (E.P. 268,616, 24.7.26).—Steel for the manufacture of edged tools contains 0·7—1·1% C, 10—16% Cr, and 0·75—2% Mn. After air-hardening, the steel is heated at 400—500° for 5—30 min. C. A. KING.

Steel alloy. POLDIHÜTTE (E.P. 245,792, 8.1.26. Conv., 8.1.25).—A steel alloy of high tensile strength and which maintains considerable resistance at high temperatures contains 4—19% W, 3·5—7% Cr, and 0·5—10% Ni.

Protection of ferrocerium. P. J. JOSEPH (F.P. 608,402, 26.12.25).—Ferrocerium rods are protected from atmospheric oxidation by coating them with an impermeable varnish or with a red-lead mixture.
A. R. POWELL.

Wear-resisting alloy. B. E. FIELD and R. FRANKS, Assrs. to HAYNES STELLITE Co. (U.S.P. 1,626,726, 3.5.27. Appl., 24.12.25).—A ferrous alloy, suitable for making articles subject to abrasion, contains 1—12% W, 25—35% Cr, 0·10—1·0% B, and 1—3·5% C.
F. G. CROSSE.

Manufacture of chromium-iron alloys. B. D. SAKLATWALLA (U.S.P. 1,623,757, 5.4.27. Appl., 5.6.26). —For the manufacture of low-carbon chrome steel, silicon is added to the metal layer of a bath of molten steel, and a mixture of chromite and chromium trioxide is added to the slag layer. The reaction between chromium trioxide and silicon evolves sufficient heat to promote reaction between silicon and chromite without additional external heating of the bath.
T. S. WHEELER.

Manufacture of magnetic alloys. V. B. BROWNE (U.S.P. 1,627,269, 3.5.27. Appl., 19.3.25).—Silicon iron of definite silicon content is melted in relatively large quantity and maintained in a molten condition until solid inclusions have floated to the surface. The process may be assisted by agitating the molten metal.
C. A. KING.

Copper-aluminium alloys. METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 268,654, 21.9.26. Conv., 15.4.26).—The strength of copper-aluminium alloys when hot is increased while still being malleable if the composition falls between the limits 5—9·5% Al, 1—6% Mn, 0—3% Ni, 0·1—3% Fe, and 0·01—2% Sn. The tensile strength of a particular alloy containing 84% Cu, 1·5% Fe, 5% Mn, 9% Al, and 0·5% Sn was 80 kg./mm.^{2} C. A. KING.

Production of zinc. F. KRUPP GRUSONWERK A.-G. (E.P. 255,482, 16.7.26. Conv., 20.7.25).—Zinciferous material is distilled until the zinc content is reduced to 10—15%. The residue is reduced and heated in a current of air to volatilise zinc oxide, which is recovered and treated in a muffled distilling furnace.
C. A. KING.

Recovery of zinc from dross obtained in melting zinc during refining. E. C. R. MARKS. From AMERICAN SMELTING AND REFINING Co. (E.P. 269,321, 7.4.26).—The dross is roasted so as to volatilise the contained chlorides of zinc and ammonia, which are collected and returned to the melting furnace. The calcined residue is leached for the further recovery of soluble zinc compounds. C. A. KING.

Metallurgical process [for treating copper ores]. W. E. GREENAWALT (U.S.P. 1,618,187, 22.2.27. Appl., 9.3.26. Cf. U.S.P. 1,572,115; B., 1926, 330).—Copper ore containing zinc is crushed and concentrated, the concentrate being roasted and leached with a dilute acid solution to dissolve copper, zinc, and iron salts. The solution, after treatment with sulphur dioxide to reduce ferric salts, which interfere with electrolysis, is electrolysed to recover copper and oxidise ferrous irou, and finally is treated with crude zinc oxide to precipitate iron and residual copper, and yield a solution from which zinc can be recovered. T. S. WHEELER.

Conversion of matte containing nickel. O. LELLEP, Assr. to INTERNAT. NICKEL Co. (U.S.P. 1,623,797, 5.4.27. Appl., 22.3.26. Conv., 7.7.26).—Matte containing nickel is blown with air in a converter to which sand is added, and to promote reaction a flame is directed on the material in the converter. When the sulphur has been reduced to a point at which oxidation of the metal begins, the air in the blast is replaced by waste gas from the converter, containing about 3% of oxygen, which completes the oxidation of the sulphur.
T. S. WHEELER.

Production of aluminium alloys having a high silicon content. T. GOLDSCHMIDT A.-G. (E.P. 255,103, 12.7.26. Conv., 10.7.25).—Substances, *e.g.*, alkali chlorides, metallic oxides, etc., known to produce alloys of aluminium and silicon having a fine granular eutectic structure are added to the mixture of aluminium and silicon compounds prior to electrolytic fusion in a fluoride bath. C. A. KING.

Solder for aluminium and its alloys. H. TOTTMAN (E.P. 269,771, 10.8.26).—The solder consists of an alloy of equal or approximately equal parts by weight of block tin, granulated tin, and cadmium. Stearic acid is a suitable flux. F. G. CROSSE.

Treatment of antimonial ores, more especially tin ores containing antimony. ZINNWERKE WILHELMSBURG G.M.B.H. (E.P. 268,278, 5.7.26. Conv., 29.3.26).—Tin ores containing antimony are reduced either previous to or simultaneously with their extraction with an acid. *E.g.*, the ore may be mixed with 5—10% of carbon, roasted, and leached with hydrochloric acid (*d* 1·16), or treated directly with acid in the presence of a ferrous or cuprous salt. C. A. KING.

Production of hafnium from its ores. SIEMENS & HALSKE A.-G., Assees. of B. FETKENHEUER (G.P. 437,352, 17.2.24).—The ore is decomposed by treatment with

chlorine and a reducing agent such as carbon, and hafnium chloride, which is subsequently worked up to hafnium, is separated from other gaseous products by diffusion. The separation may be effected with the aid of carbon tetrachloride vapour. L. A. Coles.

Washing minerals by means of liquid streams. A. France (E.P. 268,682, 22.11.26).—In a washing plant consisting of a series of launders in cascade formation, and in which the products delivered from the lower launders are returned for re-treatment, the product from the end of the uppermost launder is discharged on to a vibrating screen on which it is washed with clean water. The suspension of loam thereby formed is utilised to obtain a muddy liquor of suitable density for the washing plant. C. A. King.

Flotation apparatus. T. M. Owen and M. P. Dalton (U.S.P. 1,624,559, 12.4.27. Appl., 5.7.24).—A series of cells, each divided into impeller and elevator chambers, is arranged in line, so that all the impelling and elevating means can be mounted on a single shaft. The bottom of the head end of each impeller chamber communicates with its own elevator chamber, and the pulp is discharged from the tail end of each impeller chamber into the next elevator chamber, and means are provided to create a difference of pulp level between the inlet and outlet of the elevator chambers. B. M. Venables.

Continuously-operating ore or ore-shale converting retort furnace. A. M. Beam (U.S.P. 1,627,538, 3.5.27. Appl., 26.2.24).—In a roasting furnace a retort and flue consist of two channel members, the web of one channel fitting with the side flanges of the second to provide a longitudinal passage. A plate fitting on the flanges of the first channel forms a second similar passage. C. A. King.

Electric furnace for the manufacture of aluminium. E. R. Lauber (E.P. 256,193, 22.6.26. Conv., 30.7.25).—The bottom of a furnace for the production of aluminium by electrolysis consists of material, *e.g.*, alumina, corundum, magnesia, which is not attacked by molten aluminium. The cathode is formed by a layer of molten aluminium to which the current is led by means of graphite blocks. C. A. King.

Methods and apparatus for fusing metals by the electric arc process for welding, cutting, etc. British Thomson-Houston Co., Ltd., Assees. of I. Langmuir and P. P. Alexander (E.P. 257,955, 6.9.26. Conv., 5.9.25).—In the process of welding with a fusible electrode, the molten weld is flooded with a mixture of nitrogen and hydrogen, the hydrogen being in such proportion (about 6%) as to prevent nitrogenisation of the metal. C. A. King.

Manufacture of very thin wires. G. L. Hertz, Assr. to N. V. Philips' Gloeilampenfabr. (U.S.P. 1,627,971, 10.5.27. Appl., 22.4.25. Conv., 18.6.24).—See E.P. 235,893; B., 1926, 245.

Open-hearth furnace. E. Bosshardt (U.S.P. 1,627,425, 3.5.27. Appl., 30.6.24. Conv., 25.10.23).—See E.P. 223,870; B., 1925, 456.

Sealing metal to glass. G. B. Jonas, Assr. to N.V. Philips' Gloeilampenfabr. (U.S.P. 1,627,780, 10.5.27. Appl., 2.4.26. Conv., 11.5.25).—See E.P. 251,951; B., 1927, 80.

Ore washing installations (E.P. 254,693).—See I.

Separation of minerals etc. (E.P. 268,043).—See I.

Furnace (U.S.P. 1,624,043).—See I.

Handling material to be sintered (E.P. 265,533).—See I.

Beryllium filament (U.S.P. 1,622,604).—See XI.

XI.—ELECTROTECHNICS.

Silver accumulator. F. Jirsa and K. Schneider (Z. Elektrochem., 1927, **33**, 129—134).—A secondary cell in which the positive electrode consisted of silver peroxide, yielding silver on discharge, in an alkaline electrolyte was patented by Jungner in 1899, and was claimed to have several advantages. With a view of finding reasons for its lack of success in practice, a model accumulator of the type Ag|NaOH solution|Fe has now been examined. Positive plates consist of iron or nickel gauze on which has been pressed the active mass of spongy silver. Negative plates are prepared by smearing iron gauze with a paste of powdered iron and gum arabic. On charging, the silver is converted first into the oxide and then into the peroxide, the open circuit *E.M.F.* corresponding with these two stages being 1·17 and 1·47 volts. Using plates with 0·063 g. of silver per cm.^{2} of surface, and charging and discharging at 0·11 amp./dm.^{2}, the capacity was 269 watt-hours per kg. of active material and in a charge-discharge cycle the current efficiency was 95·5% and the energy efficiency 68·4%. Increasing the amount of active material per unit surface or using thicker plates diminished the capacity at given current density. In successive charges the *E.M.F.* decreases and the capacity falls notably. This is attributed to silver peroxide, which tends to form a colloidal suspension, being carried by cataphoresis to the negative plate, and there reduced to silver. This action does not occur if the electrolyte is kept free from carbonate, and it can be prevented completely, even when much carbonate is present, by the addition of potassium sulphate. H. J. T. Ellingham.

Electro-deposition of tin. Fraine.—See X.

Electrolytic corrosion tests. Blum and Rawdon.—See X.

Patents.

Method and apparatus for electroplating. British Thomson-Houston Co., Ltd., Assees. of C. E. Jones (E.P. 259,983, 15.10.26. Conv., 15.10.25).—In an electroplating process the anode consists of scrap metal held in a "cage" of perforated metal, *e.g.*, expanded sheet, which is itself in electrical connexion with the metal container. C. A. King.

Apparatus for the electrolysis of water. L. Casale (E.P. 268,998, 1.6.26).—The active faces of the electrodes are made of wire nets or perforated metal sheet, and one or more diaphragms, formed of asbestos cloth kept taut by a rod, are inserted between adjacent active faces of opposite polarity. These are carried by supports, each having two diverging extensions, for conveying into the respective gas-receiving bells the gases evolved between the diaphragm and the active face of the electrode. J. S. G. Thomas.

Decreasing the destruction of graphite and carbon electrodes used in the electrolysis of chlorides. KÖNIGSBERGER ZELLSTOFF-FABR. & CHEM.-WERKE KOHOLYT A.-G., and E. SCHLUMBERGER (G.P. 437,532, 30.7.24).—Electrolyte is caused to flow into the bath, wholly or in part, through pores, channels, etc. in the electrodes, the channels being of such form that the length of path travelled by the electrolyte in the electrodes is throughout the same. J. S. G. THOMAS.

Apparatus for ozonising air and converting it into nitric oxide. A. NEGLE (E.P. 269,046, 30.8.26).—A combustion device consisting of several arc-producers contained in a closed compartment is arranged above an ozoniser having the form of a surface condenser arranged in a separate air-tight closed casing. Both devices are connected to a source of high-tension, high-frequency multiphase current producing a strong electric field in the ozoniser and a large superficial homogeneous arc field in the combustion chamber.
J. S. G. THOMAS.

Cement for joining graphite plates used as electrodes. F. WIECZOREK (G.P. 437,552, 13.8.25).—Powdered grog, clay, sand, graphite, and water-glass are mixed to form a paste. The cement so produced is suitable for making electrodes used in the carbide industry, and lasts as long, at the high temperature of working, as the cemented graphite plates. J. S. G. THOMAS.

Induction furnace. M. UNGER, Assr. to GENERAL ELECTRIC Co. (U.S.P. 1,626,437 and 1,626,485, 26.4.27. Appl., [A], 31.12.25. [B], 16.2.21).—(A) A crucible forming a reservoir for the charge has a looped channel communicating therewith at its periphery and a primary winding for inducing heating currents in the liquid charge in the channel which has a greater cross-sectional dimension in a direction at right angles to its axis in order to permit increased circulation of the charge by electromagnetic repulsion. (B) A furnace body, forming a main chamber for containing a pool, has a number of open-looped channels arranged side by side on a common winding axis communicating at their ends with the chamber and with each other at their bends.
J. S. G. THOMAS.

Getter [for incandescence lamp filaments] and its application. D. MACRAE and H. K. RICHARDSON, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,623,351, 5.4.27. Appl., 8.6.22).—Incandescence lamp filaments are passed through an aqueous suspension of a getter having a refractory constituent, *e.g.*, thorium oxide, and a fusible constituent, *e.g.*, cryolite, into a tube furnace, where they are heated to fuse the cryolite and produce adherence of the getter. After being sealed into the lamp, each filament is heated to volatilise the getter.
T. S. WHEELER.

Beryllium filament. A. NYMAN, Assr. to DUBILIER CONDENSER CORP. (U.S.P. 1,622,604, 29.3.27. Appl., 7.11.24. Renewed 4.3.26).—Beryllium is claimed as the principal ingredient of the filaments of thermionic valves. T. S. WHEELER.

Removing residual gases from the metal casings of electric discharge tubes. SIEMENS-SCHUCKERTWERKE G.M.B.H. (E.P. 257,620, 27.8.26. Conv., 28.8.25).—The metal casing is surrounded by a gas or liquid, *e.g.*, liquid or gaseous sodium or potassium, which absorbs residual gases more easily than does the metal of the casing. A temperature gradient being established in the metal jacket, residual gases diffuse from the inside to the outside of the casing. Preferably, the pressure in the medium surrounding the casing is so low that the average length of free path of gases coming in contact with the casing is so great that only molecular flow controls the flow of gas, so that penetration of gas into the discharge vessel is prevented. J. S. G. THOMAS.

Electric fuse. SIEMENS & HALSKE A.-G. (G.P. 437,053, 7.10.23).—A fuse is constructed of a metal with a high conductivity, such as copper, coated electrolytically with chromium or cobalt. L. A. COLES.

Thermo-element for measuring high temperatures. F. KRUPP A.-G., Assees. of F. STÄBLEIN (G.P. 438,019, 24.6.24).—An element capable of prolonged use at 1300° has one arm constructed of a heat-resisting chromium–nickel alloy, containing, *e.g.*, 84—88% Ni and 12—16% Cr, and the other of a chromium–nickel–iron alloy with an iron content of at least 20%, containing, *e.g.*, 60% Ni, 25—30% Fe, and 10—15% Cr.
L. A. COLES.

Production of dry cells. TWIN DRY CELL BATTERY Co., Assees. of C. P. DEIBEL and W. G. WAITT (E.P. 244,494, 15.12.25. Conv., 15.12.24).

[Suspension for plates in] secondary electric batteries. "HART" ACCUMULATOR Co., LTD., and C. R. HARDY (E.P. 269,380, 28.7.26).

X-Ray tube. G. HOLST and A. BOUWERS, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,626,465, 26.4.27. Appl., 6.11.23. Conv., 6.12.22).

Electric furnace for aluminium (E.P. 256,193).—See X.

Insulating materials (G.P. 437,115).—See XIII.

XII.—FATS; OILS; WAXES.

Determination of the higher saturated fatty acids insoluble in water in fats and fatty acid mixtures. S. H. BERTRAM (Chem. Weekblad, 1927, **24**, 266—269).—An exhaustive review of the literature is given, and the sources of error in the various methods are described. Accurate results may be obtained by oxidising the saponified material with alkaline permanganate, removing the fatty acids after acidification by means of light petroleum, transforming these into the magnesium salts, those of the higher fatty acids being precipitated whilst those of all other acids remain in solution, treating the precipitate after washing with dilute sulphuric acid, and extracting the higher insoluble fatty acids with light petroleum. Results are given for mixtures made up from specially purified acids, for oleic and other unsaturated acids from various sources and after various "purifications," and for a large number of common oils and fats. S. I. LEVY.

Chemical mechanism of linseed oil drying. W. L. EVANS, P. E. MARLING, and S. E. LOWER (Ind. Eng. Chem., 1927, **19**, 640—641).—The relative effect on both the acid value and the iodine number of drying

linseed oil films to which had been added previously equimolecular amounts of lead acetate, manganous acetate, or cobalt acetate was studied by the general procedure previously described (B., 1927, 82). 0·01 mol. of the crystalline acetates was incorporated in 200 g. of linseed oil of iodine value 176, a temperature of 271° for 20 min. being sufficient to complete the incorporation, and this value was chosen as unity. Results were obtained at concentrations 0·05, 0·10, 0·20, and 0·40 of the chosen unit. From the results plotted graphically, it was found that a general inverse relationship of the iodine value and the acid value exists in a drying linseed oil film, and the concentration of the drier bears a definite relationship, of an exponential character, to the decreasing iodine value. Furthermore, the general difference in behaviour of the three driers expressing both acid values and iodine values, respectively, as functions of time, is in accord with the well-known relative difference in the drying speed of oils containing these driers. The general results obtained are in agreement with the view that during drying peroxide formation occurs at the ethenoid linkages of the unsaturated acids of the glyceride with subsequent splitting of these peroxides into aldehydes which in turn are oxidised to acids. H. M. Langton.

Tung oil from Australian grown trees of *Aleurites Fordii* (Hemsley). A. R. Penfold (Tech. Museum, Sydney, 1926, Bull. No. 12. 9 pp.).—The yield and analytical constants of oils obtained from tung oil trees grown in New South Wales are given. Although the quantity of fruit obtained is rather small, the quality of the oil is at least equivalent to Chinese oil.

S. S. Woolf.

Cockchafer oil. E. Kopp (Seifensieder-Ztg., 1926, **53**, 800).—The raw material, dried by gentle heat, yields on extraction 14·2—16·9% of a thick reddish-brown oil of characteristic, not unpleasant smell, and having d^{15} 0·925, acid value 42·5, saponif. value 157·5, ester value 115·0, Reichert-Meissl value 1·8, iodine value 73·7, n_D^{20} 1·4678. The isolated fatty acids are grey-green and soft, and have m.p. 31°, f.p. 27°. The dried residue from which the oil has been removed contains 66·9% of protein, and can be used as cattle food. S. S. Woolf.

Bromine-iodine values of fats. Vaubel.—See XIX.

Patents.

Stabilising emulsions of wool oils and water. I. G. Farbenind. A.-G., Assees. of Farbwerke vorm. Meister, Lucius, & Brüning (E.P. 246,867, 29.1.26. Conv., 29.1.25).—In the manufacture of the emulsions of oil and soap solution used for wool-softening (*e.g.*, "softening oil," "spinning oil," "spike oil"), an aromatic, hydroaromatic, or aliphatic-aromatic sulphonic acid of high mol. wt. or a salt thereof is used as protective colloid. These stabilisers have higher protective power than mucilaginous substances, do not cause the formation of hardening crusts on drying, and are readily removed in the subsequent "scouring" process (cf. E.P. 169,962; B., 1923, 22 a).

S. S. Woolf.

Emulsification of oils and fats and products made therefrom. R. Douglas and H. G. Loesch, Assrs. to Douglas Pectin Corp. (U.S.P. 1,625,641, 19.4.27. Appl., 4.9.23).—A solution of pectin is used to stabilise oil or fat emulsions. S. S. Woolf.

Manufacture of fatty acids from hydrocarbons. W. C. H. Pataky and F. J. Nellensteijn (U.S.P. 1,627,452, 3.5.27. Appl., 24.8.25. Conv., 27.8.24).—See E.P. 239,178; B., 1925, 929.

Detersive compound. J. F. Moseley (U.S.P. 1,627,446, 3.5.27. Appl., 1.9.21. Conv., 16.10.19).—See E.P. 162,691; B., 1921, 478 a.

Manufacture of perfumed medicated transparent soaps. P. Villain (U.S.P. 1,626,708, 3.5.27. Appl., 31.10.24. Conv., 7.11.23).—See E.P. 228,282; B., 1925, 251.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Painting cement and plaster: problems involved in, and the results of, super-accelerated alkali-water tests. H. A. Gardner (U.S. Paint Manufrs. Assoc., Feb., 1927, Circ. No. 299, 65—124).—An account is given of a series of tests designed to simulate the action on paint of the strongly alkaline surfaces of damp, freshly-laid surfaces of cement and plaster. Each of 8 types of paint was applied over each of 16 types of primer, on fresh cement and plaster slabs, which were subsequently placed in water tanks to about two thirds of their thickness, the water seeping through the panels from the underside and meeting the painted surface charged with alkali. Full details of the composition of the coatings applied and of the behaviour of the panels after 1, 8, and 12 days' immersion and after air-drying and heating are tabulated, and photographs of typical panels reproduced. Further exposure tests on similar panels in warm, moist air and in cold air, and an examination of the water-resisting powers of the primers used are also described. The results are discussed, and the importance of suitable priming is stressed. General suggestions for treatment of cement, stucco, and plaster surfaces, exterior and interior, are put forward.

S. S. Woolf.

Commercial flat white wall paints (lithopone type). E. F. Hickson (U.S. Paint Manufrs. Assoc., Mar., 1927, Circ. No. 305, 204—213).—The following data are tabulated for 27 flat white wall paints of the lithopone type:—chemical analysis, consistency, brushing and levelling qualities, reflection and gloss, fastness to light, elasticity (Kauri reduction tests and various stoving and bending tests), behaviour on heating painted panels for 5 hrs. at 105°, keeping qualities, and durability (roof exposure for 3 months). The results are discussed with reference to proposed changes in Federal Specification No. 21 (U.S. Government Specification for flat interior lithopone paint). A relation between elasticity tests and exposures is indicated. S. S. Woolf.

Covering power [of pigments] and degree of dispersion. F. Hebler (Kolloid-Z., 1927, **41**, 365—371).—The conclusion reached by Kühn (B., 1926, 287) that the covering power of a pigment is not affected by the size of the particles, but only by their number, has been further investigated. A number of homo-disperse systems of graphite were prepared by subjecting a

suspension of graphite to sedimentation, the coarser sediments being obtained by fractional elutriation, the finer by Perrin's method of fractional centrifuging (Koll.-Chem. Beih., 1909, 1, 251). The particle radius was determined by sedimentation analysis, using Stokes' formula, and also directly from the experimental results, using Svedberg's formula (A., 1924, ii, 96). Good agreement was obtained by the two methods. The permeability to light of the graphite suspensions was now measured on the optical bench, and it was found that for both reflected and transmitted light the absorption of light increases with increasing number of particles and with decreasing particle size (*i.e.*, increasing degree of dispersion). Kühn's results are thus not confirmed.

L. L. BIRCUMSHAW.

Chemical mechanism of linseed oil drying. EVANS, MARLING, and LOWER.—See XII.

PATENTS.

Production of printing inks. M. LUDWIGSEN (E.P. 268,966, 13.4.26).—Waste from cellulose manufacture, *e.g.*, sulphite lye or terpenes or these two substances containing small proportions of resin, is evaporated to a solid content of 40—60%, and glycerin or a glyceride (rendered viscous by the addition of oily matter, *e.g.*, wheel grease, petroleum jelly) is added. Linseed oil factice may replace part of the glycerin etc. Colouring matter is finally incorporated.

S. S. WOOLF.

Oxidised pine oil for pigments. J. K. SPEICHER, Assr. to HERCULES POWDER CO. (U.S.P. 1,626,362, 26.4.27. Appl., 20.7.26).—The pigment is ground in admixture with oxidised pine oil. H. ROYAL-DAWSON.

Production of oil-varnish substitutes. G. PETROV (G.P. 437,612, 26.3.24).—Phenols are condensed with formaldehyde in the presence of acidic or basic catalysts, and the products, after cooling to 40—50°, are mixed with naphthenic acids obtained by oxidation of naphtha or other mineral oils. The resulting semi-fluid substances are dissolved in alcohol and used as insulating varnishes, binding media for moulding compositions, etc.

S. S. WOOLF.

Preparation of condensation products of carbamide and formaldehyde. I. G. FARBENIND. A.-G., Assees. of W. GEISEL (G.P. 437,646, 18.11.24. Addn. to G.P. 416,252; B., 1926, 99).—During the curing process applied to the syrupy condensation products of carbamide or its derivatives with formaldehyde or its polymerides, hardening is accelerated and the tendency to clouding and even depositing is counteracted if the mass be subjected to the action of ultra-violet light.

S. S. WOOLF.

Treatment of condensation products of carbamide or its derivatives with formaldehyde. F. POLLAK (E.P. 266,389, 30.9.25. Conv., 1.10.24).—Hardened final condensation products of carbamide or its derivatives and formaldehyde are reduced to a fine powder mechanically or by boiling with mineral acid (of 10% strength) and cooling. The powder so obtained is softened by treatment with a small quantity of any of the solvents mentioned in E.P. 240,840 (B., 1926, 22), or of the soluble initial condensation products of the above-mentioned substances, and may then be moulded under the combined action of pressure and heat.

S. S. WOOLF.

Manufacture of resinous insulating and impregnating materials. SIEMENS-SCHUCKERTWERKE G.M.B.H. (G.P. 437,115, 2.4.25).—Small amounts of bases which do not affect the physical properties of these materials are added, in order to convert the water-soluble resinic acids into insoluble salts. The amount of base added is much less than the equivalent of the resinic acids.

J. S. G. THOMAS.

Purification of abietic acid. G. H. DUPONT (U.S.P. 1,628,535, 10.5.27. Appl., 23.4.25. Conv., 28.4.24):—See E.P. 244,980; B., 1926, 202.

Process of colouring materials. J. P. TRICKEY, Assr. to QUAKER OATS CO. (U.S.P. 1,627,939, 10.5.27. Appl., 2.1.23. Renewed 23.3.27).—See E.P. 234,862; B., 1926, 642.

Production of resinous substances. CHEM. FABR. DR. K. ALBERT, G.M.B.H., Assees. of A. AMANN and E. FONROBERT (U.S.P. 1,623,901, 5.4.27. Appl., 4.2.24. Conv., 18.8.17).—See E.P. 259,030; B., 1926, 988.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Rubber compositions. F. C. DYCHE-TEAGUE (E.P. 268,853, 10.12.25).—The mixture of carbon and catalyst formed, *e.g.*, by the decomposition of carbon monoxide in the presence of finely-divided nickel or iron, is used as a compounding ingredient for rubber. The incorporation of 36 pts. with rubber (100 pts.), zinc oxide (10 pts.), sulphur (4 pts.), and diphenylguanidine (1 pt.), after vulcanisation, yields a product superior to one otherwise similar but containing gas black, in its greater toughness and decreased deadness. The mixture of carbon and catalyst is also more compact, more easily introduced into rubber, and expedites vulcanisation. D. F. TWISS.

Manufacture of rubber products. A. R. F. VAN DER MARK and H. KREMER (Swiss P. 116,630, 8.7.25. Conv., 11.7.24).—Unvulcanised coagulated latex is shaped into hollow or sac-like form and the walls are stretched until the desired thickness is attained; the product is then dried and can be vulcanised. Colours may be introduced into the latex before coagulation.

D. F. TWISS.

Vulcanisation of rubber. C. J. T. CRONSHAW and W. J. S. NAUNTON, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,627,636, 10.5.27. Appl., 28.6.26. Conv., 9.9.24).—See E.P. 244,159; B., 1926, 138.

Manufacture of a deterioration retarder for rubber. S. M. CADWELL, Assr. to NAUGATUCK CHEMICAL CO. (U.S.P. 1,626,784, 3.5.27. Appl., 23.9.24).—See E.P. 240,407; B., 1925, 1000.

XV.—LEATHER; GLUE.

Specific ion effects in the behaviour of tanning agents towards collagen treated with neutral salts. K. H. GUSTAVSON (Fourth Colloid Symposium Monograph, 1926, 79—101).—There is at present no sharp division between primary, secondary, and residual

valencies in protein reactivity. Vegetable tannage is largely a function, under like conditions, of the state of hide powder as to secondary valency and specific surface forces. The one-bath chrome tanning process is essentially a primary valency reaction. There are three factors involved in the specific ion effects shown by very basic fresh chromium sulphate liquors with collagen treated with neutral salts: (1) a primary valency reaction between positively-charged chromium complexes or micelles and protein carboxyl groups, (2) formation of molecular compounds between complex chromium anions and basic protein groups, (3) adsorptive processes.

Chemical Abstracts.

Testing glue. Meess (Betriebsführung, 1926, 5, 120—125; Chem. Zentr., 1927, I, 677).—Rudeloff's method (cf. B., 1918, 743 a) gives reliable results. The tenacity does not decrease regularly with increasing dilution of the glue; with increases from 100 to 150% and from 200 to 300% of water, the change in tenacity is only small, but it decreases rapidly as the water is increased from 150 to 200%. The humidity of the air has but little effect upon the tenacity, which also is only affected by prolonging the drying period from 24 hrs. to 48 hrs. when the weaker glue is used. Heating the glue at 40°, but not at higher temperatures, is advantageous with thick glue. L. A. Coles.

Preservation of gelatin emulsions. T. Sabalitschka and E. Böhm (Chem.-Ztg., 1927, 51, 301—302).—Gelatin solutions were treated with various preservatives in increasing amounts up to 0·1%, and observations made of the time required for mould to appear, for the mould to form a complete covering, and for the complete breaking down and liquefaction of the jelly. Boric acid and sodium benzoate showed a lower bactericidal action than benzoic and salicylic acids, but even in the case of the latter the formation of mould was only delayed and not entirely prevented. By the addition of 0·1% of methyl *p*-hydroxybenzoate the gelatin solutions could be preserved for periods varying from 40 to 71 days without the appearance of mould, or of such turbidity or coloration as occurred in the case of the other preservatives. F. R. Ennos.

Patents.

Manufacture of shaped articles of casein. A. Bartels and O. Miech (U.S.P. 1,623,085, 5.4.27. Appl., 24.3.26. Conv., 15.5.23).—Unhardened casein material, roughly moulded to the desired shape, is partially hardened by immersion in hexamethylenetetramine solution, dried till the moisture content is reduced to about 17%, rapidly softened by the action of heat, finally formed by pressing, and completely hardened and dried. T. S. Wheeler.

Dyeing of leather. M. C. Lamb (U.S.P. 1,628,160, 10.5.27. Appl., 9.6.25. Conv., 27.4.25).—See E.P. 255,555; B., 1926, 839.

Manufacture of basic chromium salts (E.P. 251,267).—See VII.

XVI.—AGRICULTURE.

Manuring in drills. H. Burk (Z. Pflanz. Düng., 1927, B6, 145—162).—In numerous field trials with sugar beet, superphosphate gave better results when drilled in with the seed than when sown broadcast. In parallel experiments with Rhenania phosphate, basic slag, and dicalcium phosphate the reverse proved to be the case. A. G. Pollard.

Determination of the manurial requirement of soils for phosphate by means of the citrate method. O. Lemmermann, L. Fresenius, and Lesch (Z. Pflanz. Düng., 1927, B6, 163—178).—The customary determination of citric-soluble phosphate in soils is supplemented by a consideration of the "relative solubility," viz., the percentage of total phosphate which is soluble in 1% citric acid solution. Total phosphate is extracted with aqua regia and in highly organic soils preliminary treatment with nitric acid is recommended. The carbonate content of the soil is determined by means of Schiebler's apparatus, using citric acid. In preparing the citric acid extract of soils, sufficient acid is used to neutralise carbonates and give in addition a 1% solution of free acid. Soil and citric acid solution are shaken together for 6 hrs. followed by a further 2 hrs. on the subsequent day. Soils having more than 0·025% of citric-soluble phosphate do not usually need phosphate when the "relative solubility" is greater than 25%. Where the citric-soluble phosphate is less than 0·020%, a phosphate requirement is indicated. Between these two ranges the phosphate requirement is ill-defined and varies with the "relative solubility" figure. Detailed comparisons between the above method, pot experiments, field experiments, and the Neubauer method are recorded and discussed. A. G. Pollard.

Laboratory and field experiments on the use of 3 : 5-dinitro-o-cresol and its sodium salt for winter spraying. C. T. Gimingham and F. Tattersfield (J. Agric. Sci., 1927, 17, 162—180).—Observations are recorded on the use of solutions of 3 : 5-dinitro-*o*-cresol and its sodium salt as sprays against the eggs of certain insects. Both substances are effective and, in addition, have a cleansing action on the bark of trees. The concentration used in the spraying experiments on trees was a 0·25% solution of 3 : 5-dinitro-*o*-cresol, or its equivalent in the case of the sodium salt.

G. W. Robinson.

Relationship between the chemical constitution of organic compounds and their toxicity to insects. F. Tattersfield (J. Agric. Sci., 1927, 17, 181—208).—There is a rough correlation between both the mol. wt. and volatilities of organic compounds and their toxicity as contact insecticides, but the direct connexion is probably with some other property, such as adsorption. An account is given of the chemical compounds found in certain highly toxic tropical leguminous plants. The toxicity of a number of organic compounds to *Aphis rumicis* and to the eggs of *Selenia tetralunaria* is examined in relation to their chemical constitution. Substitution of certain radicals in the benzene ring affects toxicity, but the number substituted and their relative position also have an effect. 3 : 5-Dinitro-*o*-cresol has a powerful ovicidal effect. Toxicity increases in the fatty acids up to undecoic acid, after which it declines. Their physical properties only partially explain their toxic action.

G. W. Robinson.

Soil biodynamics. I. Microbiological characteristics of southern soils. S. KOSTICHEV, A. SHELOUMOVA, and O. SHUL'GINA (Bull. Bur. Agric. Microbiol. [Russia], 1926, 1, 5—45).—In these soils there are two simultaneous processes: (*a*) the nitrogen-fixing organisms are active, while other groups of micro-organisms are decomposing the native rock and supplying mineral substances; (*b*) mineralisation of organic residues increases the nitrogen content and supplies mineral constituents for the plant. In certain Crimean soils the nitrogen balance is maintained without the addition of nitrogen. CHEMICAL ABSTRACTS.

Soil biodynamics. II. Microbiological characteristics of the soils in the podsol region. 1. Fixation of atmospheric nitrogen. M. KORSAKOVA and V. BILINKINA (Bull. Bur. Agric. Microbiol. [Russia], 1926, 1, 47—72).—Cultivated soils maintain the highest number of *Azotobacter* and *Clostridium*.

CHEMICAL ABSTRACTS.

Soil biodynamics. III. Microbiological characteristics of the soils in the podsol region. 2. Energy of soil microbiological activities. M. KORSAKOVA and G. LOPATINA (Bull. Bur. Agric. Microbiol. [Russia], 1926, 1, 73—84).—No correlation was found between the energy of utilisation of mannitol and the presence of nitrogen-fixing organisms.

CHEMICAL ABSTRACTS.

Availability of phosphates in calcareous or alkaline soils. J. F. BREAZEALE and P. S. BURGESS (Arizona Agric. Exp. Sta. Tech. Bull., 1926, 10, 209—237).—Neutral or acid soils in humid regions (especially in abundance of organic matter), but not calcareous or alkaline soils in semi-arid regions, may respond to applications of insoluble phosphates. Plants are able to absorb phosphorus readily from solutions containing as little as $0 \cdot 1$ in 10^6 parts. The presence of dissolved carbon dioxide increases the solubility and availability of the phosphorus. Tricalcium phosphate alone is of little value as a source of phosphorus. The slow reaction of iron and aluminium hydroxides with calcium phosphate is accelerated by carbonic acid alone, but retarded by carbonic acid and calcium carbonate. In a neutral soil containing no carbonic acid, in a black alkali soil, or in a soil containing excess of carbonic acid and calcium carbonate, the plant will be more favoured than the iron and aluminium hydroxides in the competition for the phosphorus in applied calcium phosphate. CHEMICAL ABSTRACTS.

Determination of the colloidal material in soils. G. J. BOUYOUCOS (Science, 1926, 64, 362).—The colloidal content of a soil can be determined in 15 min. by dispersing 50 g. of soil in a mortar, placing it in a tall cylinder with a total volume of 1050 c.c. of water, shaking vigorously for 2 min., and determining the density of the liquid by means of a hydrometer. The percentage of the material remaining in suspension at the end of 15 min. is equal to the percentage of the colloids as determined by the heat of wetting method. Only 3 of 31 soils gave abnormal results. The rate of settling can also be determined by the hydrometer method. A. A. ELDRIDGE.

Rapid determination of the moisture content of soils. G. J. BOUYOUCOS (Science, 1926, 64, 651—652).—The soil is vigorously shaken with absolute ethyl alcohol, the mixture immediately filtered, and the change in density of the alcohol is determined by means of a hydrometer. The method is almost as absolute as the oven method. A. A. ELDRIDGE.

PATENTS.

Obtaining a culture mixture for fertiliser production. C. A. BAUMGARTEN-CRUSIUS (U.S.P. 1,626,579, 26.4.27. Appl., 12.2.26. Conv., 23.1.23. Cf. E.P. 258,149; B., 1926, 991).—The process comprises subjecting a small mass of decomposing soil, horse manure, and the like to a temperature of 60° for about 20 min. to destroy undesired organisms, then dividing the mass into two portions, sterilising one, dividing the other into two parts, and keeping these at a temperature favourable to the growth of bacteria, one part being under aerobic and the other under anaerobic conditions. The culture is then made by lixiviating the two parts, and adding the liquid to the sterilised portion of soil. This is then used for inoculating a mass of decomposable material consisting of soil, horse manure, beech leaves, and peat.

C. T. GIMINGHAM.

Production of fertiliser. A. G. STILLWELL (U.S.P. 1,622,684, 29.3.27. Appl., 21.10.24).—Waste-water from fish- and meat-canning factories is freed from oil, evaporated to *d* $1 \cdot 24$—$1 \cdot 41$, and the syrup thus obtained is mixed with calcium hydrogen phosphate and dried at 120°. T. S. WHEELER.

Combating injurious fungi. I. G. FARBENIND. A.-G. (G.P. 436,923, 13.2.23).—The poisonous action of thallium compounds on all types of fungi is much greater than that of other known inorganic poisons, such as mercuric chloride or cyanide. To prevent the growth of such organisms on wood, therefore, the latter may be impregnated with a solution of thallium sulphate containing $0 \cdot 2$ pt. per 1000, or a solution of another thallium salt, *e.g.*, the sulphite, oxalate, or sodium thallium thiosulphate, etc., of equivalent thallium content. A. B. MANNING.

XVII.—SUGARS; STARCHES; GUMS.

Determination of starch in confectionery. GROSSFELD.—See XIX.

PATENTS.

Preparation of [pure] tricalcium saccharate. C. STEFFEN (E.P. 261,693, 4.1.26. Conv., 17.11.25).—Tricalcium saccharate is washed under high pressure in a press, and mashed with 7—8 times as much water or lime water. Lime is added until $0 \cdot 4$—$0 \cdot 5\%$ of sugar remains in solution. The tricalcium saccharate suspension thus obtained is used for washing fresh tricalcium saccharate obtained by the precipitation of sugar from a molasses or impure sugar solution with lime powder. The washing is effected in the filter press. Tricalcium saccharate of at least 99% purity is obtained.

B. FULLMAN.

Manufacture of lactose. S. C. MEREDITH and N. N. T. NYBORG (U.S.P. 1,626,857, 3.5.27. Appl., 12.12.22).—Alkali is added to whey in excess of that

required to neutralise the acid content, the whey is then heated gradually to coagulate and precipitate the albumin, and the alkali content is gradually decreased as the temperature increases. W. G. Carey.

XVIII.—FERMENTATION INDUSTRIES.

Clarification of wine. O. Reichard (Z. Unters. Lebensm., 1927, **53**, 163—168).—The clarification of wine by means of potassium ferrocyanide is described. When the treatment is correctly performed, by adding the exact amount of ferrocyanide, the whole of the salt occurs in the dregs, which normally are removed by filtration or by settling. The wine then contains only a trace of iron in solution, and when centrifuged gives a slight deposit of Prussian blue. If the wine is "over-clarified," by adding too much of the "clearing salt," then a greenish opalescence is produced in the wine, Prussian blue is deposited on centrifuging, and free ferrocyanide and hydrocyanic acid are present in solution. This latter can be determined by making the wine strongly alkaline, warming it at 60°, and driving the hydrocyanic acid into silver nitrate solution by a stream of carbon dioxide. The correction of an excessive addition of ferrocyanide by adulteration with a wine rich in iron ("back-clarifying") is illicit, as the effects vary with the time which elapses between the "over-clarifying" and its correction. If carried out immediately, no ill-effects follow, but if the "over-clarified" wine is kept, the excess ferrocyanide decomposes and, on addition of the iron compound or of the wine containing iron, is precipitated leaving free hydrocyanic acid in solution. H. J. Dowden.

Methyl alcohol contained in spirits prepared from grape and fruit residues. G. Reif (Z. Unters. Lebensm., 1927, **53**, 168—171).—Methyl alcohol occurs in brandy from the decomposition, during fermentation, of the methyl ester of pectic acid present in the fruit skins. The author has examined the methyl alcohol content of numerous samples of grape and fruit brandies. In the manufacture of the fruit brandy the skins, stalks, etc. of apples, pears, and plums, after expression of the juice, are placed in oak casks and allowed to ferment, without addition of sugar or yeast. After fermentation, the liquid is distilled with addition of wood charcoal to absorb fusel oil etc. The refined spirit contains between 40 and 50% by vol. of alcohol. The method of manufacture has no influence on the methyl alcohol content, but, in general, that of spirit produced from the marc of grapes is lower than that of spirit from fruit. For grape brandy, the methyl alcohol forms 0·3—0·7% by vol. of the total liquid or 0·6—1·4% by vol. of the total alcohol, whilst for brandy prepared from fruits, the values are 0·4—0·9%, calculated on the total liquid, or 0·8—1·8% by vol. of the total alcohol. H. J. Dowden.

Preservative values of green and of kiln-dried hops. A. C. Chapman and C. W. McHugo (J. Inst. Brew., 1927, **33**, 294).—The preservative properties of green hops during the ripening period do not increase, but, in a few cases, even diminish slightly. During kilning the reduction of preservative power is considerable. C. Ranken.

Patents.

Recovery of glycerin from fermented molasses mash. [a], S. K. Varnes, [b], J. W. Lawrie, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,626,986 and 1,627,040, 3.5.27. Appl., [a], 14.6.22. [b], 6.6.22).—(a) Unclarified concentrated slop is divided into minute portions and "exploded" into mist in contact with a hot gaseous carrier chemically inert at the temperatures necessary for the rapid vaporisation of the glycerin, which is subsequently condensed. (b) The unclarified concentrated slop is heated and atomised under a pressure of at least 30 lb./sq. in. (approx.) in contact with a gaseous carrier at a temperature at which the glycerin is vaporised. The evolved glycerin vapours are subsequently condensed. C. Ranken.

Brewing pan heated by internal steam tubes. J. Schaefer (G.P. 437,431, 3.11.23).—The pan is fitted with a number of arched tubes lying in a common plane perpendicular to the axis of the stirrer, having their ends attached to the inner wall of the pan and connected up in a continuous network by curved tubes attached to the outer wall, these tubes being provided with means for drawing off condensed water. L. A. Coles.

Separating solids from liquids (E.P. 268,051).—See I.

Drying apparatus (E.P. 268,237).—See I.

Preparations of blood and meat juice (F.P. 609,540).—See XIX.

Extraction of cream of tartar (F.P. 579,472).—See XX.

XIX.—FOODS.

Bromine-iodine values of butter and other edible fats. W. Vaubel (Z. Unters. Lebensm., 1927, **53**, 151—154).—The author has shown (cf. B., 1926, 887) that bromine values of fat can be classified as primary and secondary. A tertiary value is now developed, this being the absorption of bromine by the fat or oil after contact with bromide-bromate solution for 2 days. For linseed oil, these three values were 127, 172, and 240 respectively. Butter-fat values varied between 36 and 49 for the primary, 51—70 for the secondary, and 70—100 for the tertiary. The saponified fats, however, showed distinctly lower values in all three cases. Olive oil has higher values for the saponified oil than for unsaponified. The iodine values of tallow show a decrease when the fat is saponified, whilst lard shows an increase. For a series of margarine samples, the values for the saponified fat, were invariably higher than those for the unsaponified, and it is suggested that this fact affords a means of distinguishing between butter and margarine. H. J. Dowden.

Relation between the fat content of milk and that of the resultant cheese. S. Goy (Z. Unters. Lebensm., 1927, **53**, 160—163).—It is shown that the food regulation, which requires in cheese a minimum fat content of 40% in the dry material, is not too stringent. Formerly no fat limit was stipulated, but "whole" milk had to be used. Figures have been collected over a period of 1 year, for over 100 samples of cheese from

39 different factories, showing the fat content of the original milk (determined at the factory) compared with that of the cheese (determined by the author). The lowest milk-fat figure of 2·4%, which is extremely low for a mixed milk, gave a cheese of fat content 42·28%. Normal "whole" milk, of fat content 2·7% and over, produced cheese of 47·8% fat content. The whole series shows that normal milk will always give cheese which will satisfy the requirements of the food regulations.

H. J. Dowden.

Polarimetric determination of starch in confectionery products. J. Grossfeld (Z. Unters. Lebensm., 1927, **53**, 156—160).—The author shows that the method previously described (cf. A., 1917, ii, 223) is particularly suitable for the determination of starch in such materials as peach and apricot kernels used for the manufacture of marzipan. The method of heating at 3½ atm. pressure and converting the starch into dextrose by boiling with 25% hydrochloric acid involves considerable errors owing to the conversion of hemicelluloses into sugars. In the polarimetric method, to compensate for the 3% error introduced by insoluble matter and fat, the conversion factor in the formula is reduced from 5·444 to 5·28, and to compensate for sugar 0·18 is deducted from the difference in polarimeter readings. The formula then becomes:—Starch $= (B - A - 0{\cdot}18) \times 5{\cdot}28\%$.

H. J. Dowden.

Determination of the vegetable constituents of marmalade. W. Reidemeister (Z. Unters. Lebensm., 1927, **53**, 81—100).—10 g. of the material under investigation are macerated with 200 c.c. of warm water, and, after settling, the supernatant liquid and diluted pulp are decanted, and examined under the microscope. Pips, skins, and hairs of a variety of fruits and berries are encountered, and even mites, which indicate that dried fruit has been used in the preparation of the preserve. The sediment is examined macroscopically. The author describes and illustrates photographically the characteristic structure, dimensions, and behaviour towards stains, of the epidermis, seeds, sacs, styles, etc. of a large number of fruits, berries, and vegetables, which are commonly found as adulterants.

H. J. Dowden.

Storage of oranges. E. Bottini (Annali Chim. Appl., 1927, **17**, 129—163).—Oranges keep best when stored in crates or small barrels in presence of ammonium carbonate or trioxymethylene or, preferably, the two together. Fruit stored in this way is unaltered in composition, but the superficial parts undergo appreciable hardening, owing to hydrolysis of the soluble pectin by the ammonia and the consequent formation of insoluble pectic substances (pectic acid), which intensify the connexion between the cells. The retardation of the evaporation of water from the fruit, thus effected, results in a drier atmosphere, and hence acts unfavourably on the development of micro-organisms. Moreover, methyl alcohol is liberated and undergoes oxidation to aldehyde, and then to formic acid, which also have preservative influence.

T. H. Pope.

Determination of amino-acids in foodstuffs. J. Tillmans and J. Kiesgen (Z. Unters. Lebensm., 1927, **53**, 126—131).—The first method consists in titrating with caustic soda to p_H 11·8 using tropæolin O as an indicator, and as a colour standard a buffer solution of pure caustic soda containing 0·00668 c.c. of N-caustic soda per c.c. and made normal in sodium chloride. The second method is based on the observations of Willstätter and Waldschmidt-Leitz (cf. A., 1922, ii, 169), that amino-acids can be determined alkalimetrically in strong alcoholic solutions. 5—10 g. of the material are dissolved in 250 c.c. of hot water, 0·3 g. of barium chloride is added, approximately neutralised, and then 10 c.c. of $2N$-caustic soda solution are added. Ammonia is removed by aspirating air for 2½—3 hrs., after which the solution is approximately neutralised with hydrochloric acid and diluted to 100 c.c. with $2N$-sodium chloride solution, first treating with animal charcoal if the solution is dark coloured. As a comparison, determinations were made by Grünhut's formaldehyde method (cf. B., 1919, 788 A). For the tropæolin titration Grünhut's colorimeter is used (*loc. cit.*). Into one cylinder are introduced 30 c.c. of the solution, about 1·8 g. of sodium chloride to make it about normal, and 0·5 c.c. of a 0·1% aqueous solution of the indicator. Into the comparison cylinder are poured 30 c.c. of the buffer solution and 0·5 c.c. of the indicator. The amino-acid solution is titrated with $0{\cdot}1N$-caustic soda solution to the same tint as the buffer. The free alkali required to produce p_H 11·8 is titrated with $0{\cdot}1N$-hydrochloric acid solution using neutral red as indicator, and a buffer solution of p_H 7 as colour standard. The volume of free alkali is deducted from the amino-acid titration. For the alcoholic titration, 5 c.c. of acid and sodium chloride solution are mixed with 50 c.c. of 96% alcohol previously neutralised with $0{\cdot}1N$-caustic soda solution, and, after adding 0·5 c.c. of 4% alcoholic thymolphthalein solution, the mixture is titrated with $0{\cdot}1N$-caustic soda solution. A further 5 c.c. of the amino-acid solution are titrated to neutrality with either caustic soda or hydrochloric acid, using neutral red as indicator, the titer being added to, or subtracted from, the first titration according as acid or alkali is required to produce neutrality. The results obtained by all three methods were found to be in close agreement.

H. J. Dowden.

Formaldehyde titration as a means of distinguishing artificial from natural foodstuffs. J. Tillmans and J. Kiesgen (Z. Unters. Lebensm., 1927, **53**, 131—137).—A solution of the substance under investigation is treated with caustic soda solution using phenolphthalein as indicator, until the solution is rose-coloured. To a 40% solution of formaldehyde containing a little phenolphthalein caustic soda solution is also added until the red colour appears. On mixing the two red solutions, the colour remains unchanged if an artificial material is present, or only one or two drops of caustic soda solution are required to restore the colour. When natural foodstuffs are under investigation, however, the colour at once vanishes on mixing the two red liquids and a more or less considerable volume of $0{\cdot}1N$-caustic soda solution is required before the colour is restored. In the investigation of lemon juice, 10 c.c. of juice required over 100 c.c. of alkali to produce a coloration, and after addition of the formaldehyde solution

a further 2 c.c. were required to restore the colour. A sample of natural juice which had been kept for 10 years required 83 c.c. of alkali to give the red tint and 6·15 c.c. to restore it. Honey 20 g. dissolved in 100 c.c. of water required 1—3 c.c. of alkali to restore the red colour after the addition of the formaldehyde solution. One sample which gave a figure less than 1·0 c.c. was concluded to be a mixture of pure and artificial honey. Numerous samples of vinegar were examined. For the natural product, the volume of 0·1*N*-caustic soda required for the restoration of the colour varied from 2 to 8 c.c., whilst for artificially prepared vinegars the colour remained unchanged. H. J. Dowden.

Cockchafer oil. Kopp.—See XII.

Patents.

Cooling of sterilised milk or other liquid in bottles. T. D. Botterill (E.P. 268,907, 26.1.26).—The bottles, contained in crates, are passed by means of an endless conveyor, through tanks having successively lower temperatures. F. R. Ennos.

Preparation of milk chocolate. Gruyère Usines Laitières S.A. (F.P. 611,731, 25.2.26).—Milk, the fat content of which has been artificially increased, is pasteurised and evaporated *in vacuo* at a moderate temperature. The solid product, containing very little water, is powdered, mixed with the ordinary chocolate constituents, and worked into blocks etc. F. R. Ennos.

Process for making cheese. M. G. J. Fouassier (E.P. 268,889, 11.1.26).—A homogeneous cheese that is not "cooked" before ripening is prepared by crumbling the drained curds and turning them over from time to time during the ripening process. The ripened cheese is then melted and pasteurised or even sterilised by heating at 75—100° in the presence of 2% of sodium tartrate, precautions being subsequently taken to preserve it in an aseptic condition. F. R. Ennos.

Preparing fish foods. C. Birdseye (E.P. 257,222, 8.3.26. Conv., 24.8.25).—The flesh of fresh fish, reduced to fragments, is placed in moulds, with or without the addition of a small amount of salt to express the protoplasm from the cells. While cold, the fish is subjected to pressure or stirring; it finally solidifies by the aid of the retained gluey content. F. R. Ennos.

Preparations of blood and meat juice. L. A. Paret (F.P. 609,540, 20.1.26. Conv., 22.1.25).—Fresh blood or raw meat juice, after treatment with aromatic substances, is allowed to ferment and the liquid, containing all the natural vitamins, is filtered, mixed with salt, and evaporated at a low temperature. The powdered product is used as a foodstuff by mixing with cooked flour, chocolate, etc., whilst the filtered liquid forms a drink. F. R. Ennos.

Process for obtaining decomposition products from materials containing protein. E. Jena (E.P. 247,944, 10.2.26. Conv., 17.2.25).—Natural animal-covering substances, *e.g.*, hair, wool, horn, feathers, etc., consisting chiefly of keratin, are cleaned and freed from fat by dilute sodium carbonate solution, dried, and ground. The powder is hydrolysed by heating at 110° with alkali under pressure to a glue-like mass, from which an acid protein substance is precipitated by addition of acid. The filtrate on treatment with excess of alkali yields an alkaline protein substance. The products are purified by re-precipitation, and washed with water under pressure. F. R. Ennos.

Removing sulphur dioxide and compounds from food products. L. Rosenstein (U.S.P. 1,623,070, 5.4.27. Appl., 18.3.26).—The fruit is treated, either by dipping or spraying, with an aqueous solution of hydrogen peroxide containing sodium bicarbonate and allowed to dry, the adhering solution oxidising the sulphur dioxide to sulphuric acid. The strength of the hydrogen peroxide solution is adjusted according to the amount of sulphur compound to be removed. F. R. Ennos.

Manufacture of condensed milk. J. R. Hatmaker (U.S.P. 1,626,818, 3.5.27. Appl., 6.4.26. Conv., 5.11.25).—See E.P. 254,941; B., 1926, 766.

Preservation of food for men or animals. T. Sabalitschka (U.S.P. 1,627,342, 3.5.27. Appl., 8.4.25. Conv., 17.4.24).—See F.P. 596,843; B., 1926, 383.

Fertiliser (U.S.P. 1,622,684).—See XVI.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Australian synthetic menthol. A. R. Penfold (Chim. et Ind. [Compt. rend., 5th Congrès Chim. Ind., 1925], 1927. 2 pp.).—A review of the preparation and properties of synthetic menthol made by the electrolytic reduction of piperitone, with a note on the relative toxicities of synthetic and natural menthols (cf. B., 1922, 78 A; 1924, 926). E. H. Sharples.

[Determination of] mercury in mercuric salicylate. I. Griffith and P. P. Ramanuskas (Amer. J. Pharm., 1927, **99**, 242—244).—The proposed method consists in dissolving 2·5 g. of the sample in 25 c.c. of nitric acid, heating the solution, and cautiously adding 20 c.c. of sulphuric acid. When the reaction subsides, the liquid is boiled until nearly colourless, a further 10 c.c. of nitric acid are added, and the boiling is continued until the yellow colour disappears. The cooled solution is diluted to 250 c.c., and mercury determined either volumetrically by adding ferric ammonium sulphate and titrating with 0·1*N*-potassium thiocyanate solution to a permanent yellowish-red colour, or gravimetrically by precipitation as mercuric sulphide. W. J. Powell.

Italian petit-grain oils. M. A. Albricci (Parf. moderne, 1926, **10**, 252—253; Chem. Zentr., 1927, I, 655).—Oils from the leaves and young twigs of bergamot, lemon, mandarin, and bitter-orange trees grown in Calabria had the following characteristics. *Bergamot:* d^{15} 0·8991, α_D −5°, saponif. value 199·73, acetyl value 213·69, acid (as acetic acid) 0·12%, esters (as linalyl acetate) 69·9%, free alcohols (as linalol) 3·8%, citral 2·10%, constituents containing oxygen 75·8%, non-volatile residue on the water-bath 4·18%, solubility in

70% alcohol 1 in 3 vol., yield of oil 0·34%. *Lemon*: d^{15} 0·8749, α_D +23·31°, saponif. value 28·87, acetyl value 130·10, citral 26·90%, esters (linalyl acetate) 10·12%, non-volatile residue 7·53%, solubility in alcohol at 85° 1 in 8 vol., yield of oil 0·143%. *Mandarin*: d^{15} 0·9775, α_D +12°, saponif. value 179·20, methyl anthranilate 58·95%. *Bitter orange*: d^{15} 0·8971, α_D − 6·12°, saponif. value 214·6, esters (linalyl acetate) 75·10%, citral 0·49%, non-volatile residue 1·99%, solubility in alcohol at 80° 1 in 1·3 vol., yield of oil 0·204%.

E. H. SHARPLES.

Pulegone oil. P. LIOTTA (Riv. Ital. delle essenze e profumi, 1926, **8**, 103; Chem. Zentr., 1927, I, 655).—Oils from the fresh and dried plants had: d^{15} 0·9394 and 0·9389, α^{20} + 26° 6′ and + 26° 5′, acid values 0·30 and 0·31, esters (as menthyl acetate) 0·90% and 0·95%, free alcohols (as menthol) 9·25% and 9·12%, saponif. values 237 and 242, ketones 68·75% and 70% and both were soluble in 2 vols. of 70% alcohol.

E. H. SHARPLES.

Acetone oils. H. SUIDA and H. POLL (Z. angew. Chem., 1927, **40**, 505—507).—Yellow acetone oil, b.p. 60—250°, was purified by washing with 5—10% hydrochloric acid and subsequent distillation over lime, thereby undergoing an 8% loss. The resulting mixture was fractionated under reduced pressure, the fractions being collected at intervals of 3°. By formation of the bisulphite additive products, the methyl ketones were separated from the higher ketones. Further fractionation or investigation of the oxidation products enabled the constituents of those fractions of b.p. 60—152° to be identified as: acetone, methyl ethyl ketone, γ-methylbutan-β-one, pentan-β-one, δ-methylpentan-β-one, γ-methylpentan-β-one, hexan-γ-one, hexan-β-one, mesityl oxide, β-methylhexan-γ-one, γ-ethylpentan-β-one, γ-methyl-Δ^{γ}-hexen-ε-one, and heptan-β-one. Distillation of synthetic mixtures of the calcium salts of butyric and acetic acids showed that mixed ketones as well as the symmetrical compounds result. The bearing of these facts on the origin of the unsymmetrical ketones in the wood-distillate is discussed. No evidence for the existence of cyclic ketones in the heavy acetone oil was obtained (cf. Pringsheim and Bondi, A., 1925, i, 1072).

L. M. CLARK.

Essential oils of *Eriostemon Coxii* (Mueller) and *Phebalium Dentatum* (Smith). A. R. PENFOLD (J. Proc. Roy. Soc. N.S.W., 1926, **60**, 331—344).—The leaves and terminal branchlets were steam-distilled in each case and the pale yellow, mobile oils, having an odour resembling that of the passion fruit (*Passiflora edulis*) had the following characteristics. *Eriostemon Coxii*: d^{15}_{15} 0·8794—0·8810, α^{20}_D + 20·75° to + 22·6°, n^{20}_D 1·4600—1·4637, ester value 94·1—116·6, ester value after acetylation 120·0—133·5, solubility in 80% alcohol by wt. 1 in 3½—8½ vol. The average yield of oil was 0·55%, and the principal constituents were *d*-α-pinene, an olefinic terpene (ocimene), butyl *iso*valerate, amyl *iso*valerate, linalol (?), geraniol, citronellol, and darwinol, both free and combined as *iso*valerates and caproates, sesquiterpene (cadinene), with small quantities of sesquiterpene alcohol, phenolic substances, and a paraffin of m.p. 64—66°. *Phebalium Dentatum*: d^{15}_{15} 0·8704—0·8776, α^{20}_D + 18·5° to + 20·25°, n^{20}_D 1·4626—1·4666, ester value 64·4—90·3, ester value after acetylation 114·1—129·2, solubility in 80% alcohol 1 in 6—7 vol. The average yield of oil was 0·21% and the principal constituents were *d*-α-pinene, an unidentified terpene, butyl and amyl butyrates and *iso*valerates, geraniol and citronellol free and as butyrate, caproate, and formate, with small quantities of citral, sesquiterpene, sesquiterpene alcohol, phenolic substances, and a paraffin of m.p. 65—66°.

E. H. SHARPLES.

Germicidal values of some Australian essential oils and their pure constituents, together with those for some essential oil components and synthetic substances. IV. A. R. PENFOLD and R. GRANT (J. Proc. Roy. Soc. N.S.W., 1926, **60**, 167—170; cf. B., 1926, 804).—Rideal–Walker coefficients and the physical constants of Australian and Indian sandalwood oils, oil from *Zieria macrophylla*, zierone, *iso*menthol, phloroacetophenone dimethyl ether, hydrocinnamaldehyde, hydroxycitronellal, and a range of aliphatic aldehydes and alcohols of the C_8—C_{12} series are given. The aldehydes and alcohols have relatively high coefficients, *i.e.*, 1% suspensions in 7½% rosin soap solution and in alcohol gave the following respective values; C_8-aldehyde, 16 and 22; C_8-alcohol, 25 and 26. These values decreased with increase in the number of carbon atoms. The dispersion varied according to the medium used, some being more highly dispersed in alcohol than in soap solution, and *vice versa*. The germicidal effect of the alcohol itself is negligible with those substances having coefficients over 4.

E. H. SHARPLES.

Use of 3 : 5-dinitro-*o*-cresol for spraying. GIMINGHAM and TATTERSFIELD.—See XVI.

Toxicity of organic compounds. TATTERSFIELD.—See XVI.

PATENTS.

Manufacture of aromatic derivatives of formamide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 269,302, 13.3.26).—Arylformamides are produced by the action of carbon monoxide (or gases rich in it) on primary or secondary aromatic amines, at high pressures and temperatures of 70—200°, in the presence of catalysts such as formic acid, formates, formic esters, formamide or its derivatives, alkali metals or their hydroxides or alkoxides, etc. *E.g.*, formanilide is formed by the action of carbon monoxide at 200 atm. pressure on aniline, at 180—200°, in the presence of sodium and methyl alcohol.

B. FULLMAN.

Manufacture of alkyl formates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 268,890, 11.1.26).—Gas mixtures containing 40—45% of carbon monoxide (*e.g.*, water-gas), preferably freed from moisture, carbon dioxide, iron carbonyl, and sulphur compounds, are passed under pressure into an aliphatic alcohol at a suitable temperature. The residual gas, containing only 20% or less of carbon monoxide, is suitable for further reactions, *e.g.*, production of methyl alcohol, catalytic hydrogenations, ammonia synthesis, the remainder of the monoxide being removed in the usual manner if desired. Water-gas, for example, is washed

with water and alkali and passed through anhydrous methyl alcohol at 200 atm. pressure to remove moisture, and then through pressure vessels at 70° containing methyl alcohol and a little sodium methoxide, to give methyl formate. The gas may be distributed through the alcohol by means of a perforated flat spiral pipe or a perforated false bottom. Alternative forms of apparatus are described. The vessels may be tin-lined or enamelled, but copper, iron, and nickel are unsuitable. *n*-Butyl formate is similarly prepared. The matter of E.P. 252,848, and 254,819 (B., 1926, 646, 721) is disclaimed.
C. Hollins.

Conversion of alkaline-earth formates [into oxalates]. O. P. C. Bredt, Assr. to Trojan Powder Co. (U.S.P. 1,622,991, 29.3.27. Appl., 7.4.21. Renewed 12.1.24).—Calcium formate is treated with sodium hydroxide solution, and the sodium formate obtained is converted by heat into sodium oxalate, which is transformed into calcium oxalate by treatment with calcium hydroxide from the first stage of the process.
T. S. Wheeler.

Preparation of solid, water-soluble, basic aluminium acetate and formate. Chem. Fabr. Grünau Landshoff & Meyer A.-G., and E. Franke (G.P. 437,637, 16.10.21).—Aqueous solutions of aluminium acetate or formate are evaporated in the form of finely-divided sprays. Even at 130° completely water-soluble products are obtained. C. Hollins.

Preparation of benzoic acid and benzoates. G. Blanc (F.P. 586,383, 7.8.24).—Benzaldehyde is added gradually to a dry, well-stirred mixture of a catalyst (silver, platinum, nickel, copper, nickel oxide, cobalt oxide, or salts of these) and a carrier (sand, kieselguhr, powdered carbon, or anhydrous salts, such as sodium sulphate, chloride, or carbonate), air or oxygen being passed in at the same time. The temperature rises rapidly and is maintained at 100°. The powdery product is dissolved in water, and a little permanganate is added so long as decolorisation occurs. The mixture, neutralised with sodium carbonate, gives a quantitative yield of sodium benzoate on evaporation. In the example given, nickel carbonate and anhydrous sodium carbonate are used. C. Hollins.

Decolorisation and purification of lactic acid. H. Gouthière (F.P. 582,402, 6.9.23).—Nitric acid (40%) is slowly added to crude lactic acid at 85—90°, the temperature not being allowed to exceed 105°. Powdered charcoal is added to complete the decolorisation and to remove the last traces of nitric acid. The lactic acid and charcoal are filtered off through a press and separated by dissolving in water. C. Hollins.

Extraction of citric acid. F. Tallada (F.P. 570,557, 5.9.23. Conv., 13.12.22).—The juice of citrons, acid oranges, green mulberries, and other acid fruit is boiled for a short time with zinc or a zinc salt. The precipitated zinc citrate is dissolved in very dilute citric acid and electrolysed, giving zinc, oxygen, and a more concentrated citric acid solution. The latter is electrolysed again with more zinc citrate until a saturated citric acid solution is obtained, which is then evaporated. The zinc citrate may alternatively be dissolved in very dilute mineral acid before electrolysis. C. Hollins.

Purification of salicylic acid. Deutsche Gasglühlicht-Auer-Ges. m.b.H. (G.P. 437,924, 29.11.24).—On addition of an aqueous solution (*e.g.*, 5%) of a soluble salt of thorium, zirconium, or the cerite- or yttria-earth metals to crude sodium salicylate solution (containing, *e.g.*, 10% of salicylic acid), the impurities are precipitated with the first portions of the rare-earth salicylate. The bulk of the salicylic acid is obtained pure by acidifying the filtrate. The rare earth is dissolved out of the impure precipitate with the calculated quantity of dilute acid, and used again. C. Hollins.

Production of alkyl- and aralkyl-resorcinols. H. Hirzel, Assee. of W. Schilt (E.P. 256,225, 24.7.26. Conv., 30.7.25).—An aldehyde and a ketone, one of which contains a 2 : 4-dihydroxyphenyl group, are condensed together in presence of 10% sodium hydroxide, and the resulting unsaturated ketone is reduced. The hydroxyl groups may be protected by alkylation or acylation. The reaction is particularly useful for the synthesis of 2 : 4-dihydroxy-1-hexylbenzene, m.p. 65—66°, b.p. 185—188°/12 mm., from 2 : 4-diethoxyacetophenone by condensation with *n*-butaldehyde or crotaldehyde, followed by reduction with hydrogen and nickel to 2 : 4-diethoxyphenyl amyl ketone, further reduction by Clemmensen's method to 2 : 4-diethoxy-1-hexylbenzene, and de-ethylation with hydrobromic–acetic acid. C. Hollins.

Extraction of cream of tartar. V. Popoff (F.P. 579,472, 8.3.24).—The grape residues (marc of grapes) are treated, one part with alkali (preferably sodium carbonate), the rest with acid (preferably sulphuric acid). To the acid portion potassium chloride is added, and the acid and alkaline portions are stirred vigorously together. Cream of tartar, 95—97% pure, separates in good yield.
C. Hollins.

Preparation of monomethylarylamines. E. Merck Chem. Fabr., Assees. of H. Maeder and W. Krauss (G.P. 437,975, 13.9.24).—*N*-Methylenearylamines, or their polymerisation products, are reduced with the calculated quantity of hydrogen in presence of a metal catalyst at ordinary temperature. Thus *N*-methylene-*p*-aminophenol is reduced in alcoholic solution with hydrogen and palladised carbon, and the acidified filtrate is evaporated under reduced pressure to give a salt of *N*-methyl-*p*-aminophenol. Methylaniline is obtained in almost quantitative yield by catalytic reduction of anhydroformaldehyde-aniline in alcoholic solution. C. Hollins.

Preparation of ββ′-dihalogeno-ethers. W. Schoeller (G.P. 437,159, 8.7.23).—Olefines are shaken, preferably under pressure, with a concentrated aqueous suspension of mercuric sulphate; the resulting dimercurated products are converted into mercurihalogen compounds, which are finally treated with halogen in a non-hydrolysing solvent. An aqueous suspension of mercuric sulphate, shaken vigorously with ethylene under pressure in a vessel previously evacuated, absorbs the gas very rapidly, and on addition of aqueous potassium iodide to the filtered liquid ββ′-*bisiodomercuridiethyl ether* is obtained in 95% yield, with a little β-*hydroxy*-β′-*iodomercuridiethyl ether*. By the action of iodine in warm

ethyl acetate the product is converted into ββ'-*di-iodo-diethyl ether*, b.p. 93—94°/1 mm. The dichloro- and dibromo-derivatives are similarly prepared. Propylene or higher olefines may also be used. C. HOLLINS.

Manufacture of guaiacol. I. GUBELMANN, H. J. WEILAND, and O. STALLMANN, Assrs. to NEWPORT CO. (U.S.P. 1,623,949, 5.4.27. Appl., 30.9.25).—A solution of diazotised *o*-anisidine in aqueous sulphuric acid is added, with normal sodium phosphate solution to boiling copper sulphate solution. Guaiacol is obtained in 95% yield. T. S. WHEELER.

Preparation of thebaine derivatives. C. H. BOEHRINGER SOHN, CHEM. FABR., Assees. of SCHÖPF (G.P. 437,451, 12.7.24).—Thebaine, dihydrothebaine, or their salts are treated in presence of alcohols with nitrosating reagents (amyl nitrite, methyl nitrite, nitrosyl chloride, nitrososulphonic acid, etc.). To thebaine hydrochloride, suspended in a mixture of absolute alcohol, chloroform, and amyl nitrite at 0°, 10% alcoholic hydrogen chloride is added dropwise. After dilution with water and extraction with ether, the aqueous layer is separated and made alkaline with 2*N*-sodium hydroxide. A *sodium* salt crystallises out at 0°, and is decomposed with carbon dioxide, giving a new *oxime*, $C_{22}H_{28}O_5N_2$, m.p. 230—234°. From the filtrate from the sodium salt, ether extracts an alkali-insoluble *by-product*, and treatment with carbon dioxide then precipitates an isomeric *oxime*, m.p. 238—240° (decomp.). Ethyl nitrite or nitrosyl chloride gives the same two oximes, the formation of which seems to proceed according to the equation: $C_{19}H_{21}O_3N + NOCl + 2EtOH = C_{22}H_{28}O_5N_2 + H_2O + MeCl$. If methyl alcohol be substituted for ethyl alcohol, there is obtained an *oxime*, $C_{20}H_{24}O_5N_2$, m.p. 240—242° (decomp.). Dihydrothebaine, m.p. 160—161°, gives with chloroform, absolute methyl alcohol, amyl nitrite, and methyl-alcoholic hydrogen chloride, a resinous *oxime* (*hydrobromide*, m.p. 240—245°; *methiodide*, m.p. 235—240°). The products have therapeutic application. C. HOLLINS.

Preparation of derivatives of 1-phenyl-2:3-dimethyl-5-pyrazolone. I. G. FARBENIND. A.-G., Assees. of K. BÖTTCHER and F. STOLZ (G.P. 436,519, 16.5.23. Addn. to G.P. 423,028; B., 1926, 611).—Derivatives of the type (I)

$$\begin{array}{l} CMe{=\!=}C\cdot NRR' \\ | \qquad\qquad | \\ NMe\cdot NPh\cdot CO \end{array}$$

are obtained by the action of alkylating, hydroxyalkylating, or aralkylating agents upon compounds (I) in which R' = H, R being alkyl or aralkyl. The methylation of 4-benzylamino- and the benzylation of 4-methylamino-derivatives are excluded. *Compounds* (I) are described in which $R = CH_2Ph$, $R' = Et$, m.p. 62—64°; R=Me, R' = allyl, m.p. 53—54°; $R = CH_2Ph$, R' = allyl, m.p. 58—60°; $R = R' = CH_2Ph$, m.p. 92—93°; $R = Me$, $R' = CH_2\cdot CH_2OH$, m.p. 85—87° (using ethylenebromohydrin); $R = CH_2Ph$, $R' = CH_2\cdot CH_2\cdot OH$, base oily, hydrochloride, m.p. 178° (using ethylene oxide); R = benzyl, $R' = CH_2\cdot CH(OH)\cdot CH_2\cdot OH$, base oily, hydrochloride, m.p. 128—130° (using glycide); R = methyl, $R' = CO_2Et$, m.p. 119—120° (using ethyl chloroformate). The products are antipyretic, and are less toxic than the 4-benzylmethylamino-compound. C HOLLINS.

Making therapeutic materials. E. SILTEN (U.S.P. 1,626,562, 26.4.27. Appl., 5.12.24).—Derivatives of 2-arylquinoline-4-carboxylate anhydrides are dissolved in a solution of hexamethylenetetramine in proportions such that the product contains more than 1 mol. of hexamethylenetetramine per mol. of anhydride. Products for intravenous injection are made from a solution of a hexamethylenetetramine derivative (*a*) of 2-arylquinoline-4-carboxylic acid containing 6 mols. of amine per mol. of acid, and (*b*) of phenylcinchophenic acid, molar ratio as above, and having a ratio water : hexamethylenetetramine = 73 : 27 by wt. B. FULLMAN.

Acylamidohydroxyphenylarsenious oxides. I. G. FARBENIND. A.-G., Assees. of L. BENDA (U.S.P. 1,622,271, 29.3.27. Appl., 19.6.25. Conv., 3.7.24.—5-Chloro-3-acetamido-4-hydroxyphenylarsinic acid is readily reduced, by treatment with sulphur dioxide in presence of concentrated hydrochloric acid to which sodium iodide has been added, to 5-*chloro-3-acetamido-4-hydroxyphenylarsenious oxide*, which is of therapeutic value. T. S. WHEELER.

Manufacture of a diuretic product. P. CACCIA (U.S.P. 1,617,701, 15.2.27. Appl., 7.8.25).—A mixture of secretin and amylase in dilute hydrochloric acid is treated with a small quantity of toluene or sodium fluoride, and heated at 40° for 1 day. The filtered solution is of use as a hypodermic injection. T. S. WHEELER.

Preparation of cucurbocitrin. I. S. BARKSDALE (U.S.P. 1,626,321, 26.4.27. Appl., 8.7.26).—Powdered water-melon seeds are treated with slightly alkaline water at 60°. The liquid is concentrated, treated successively with basic lead acetate and hydrogen sulphide, and filtered. The filtrate is treated with ether and dry alcohol in excess, the resultant precipitate being collected and dried. B. FULLMAN.

Production of crystallised perfumes. G. JAKOVA-MERTURI (F.P. 610,673, 5.2.26).—Tartaric acid and either magnesium, potassium, or sodium chloride or a mixture of the three salts are added to the perfume alone or in alcoholic solution, and then a second alcoholic solution of the perfume containing an alkali carbonate is added. *E.g.*, sodium bicarbonate is added to both solutions, and to the mixture is added a concentrated solution of sodium or potassium silicate. E. H. SHARPLES.

Simultaneous synthesis of methyl alcohol and liquid hydrocarbons. G. PATART (E.P. 247,932, 28.1.26. Conv., 23.2.25).—See F.P. 594,121; B., 1927, 346.

Synthetic production of higher alcohols. Simultaneous manufacture of methyl alcohol and liquid hydrocarbons by synthesis. G. PATART (E.P. 247,177—8, 28.1.26. Conv., 7.2.25).—See F.P. 593,648—9; B., 1927, 347.

Reduction of halogenated hydrocarbons. A. F. S. BELLONE, Assr. to SOC. CHIM. DES USINES DU RHÔNE (U.S.P. 1,627,881, 10.5.27. Appl., 28.1.24. Conv., 21.11.23).—See E.P. 225,174; B., 1925, 613.

Preparing a reaction product of acetaldehyde and aniline. S. M. CADWELL, Assr. to NAUGATUCK

CHEMICAL Co. (U.S.P. 1,627,230, 3.5.27. Appl., 26.5.23). —See E.P. 216,478; B., 1924, 848.

Manufacture of metal iodide–alkali iodide compounds of therapeutic value. H. TRUTTWIN (U.S.P. 1,627,359, 3.5.27. Appl., 28.6.23. Conv., 3.7.22).—See G.P. 397,828; B., 1924, 967.

Production of a chloride capable of reaction from pinene or purified turpentine oil. F. HÖHN (E.P. 269,624, 11.1.26).—See G.P. 426,865; B., 1927, 378.

Mixtures for generating carbon dioxide (G.P. 437,113).—See VII.

Preparation of [pure] tricalcium saccharate (E.P. 261,693).—See XVII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Law of blackening of the photographic plate at low densities. E. A. BAKER (Proc. Roy. Soc. Edinburgh, 1926, **47**, 34—51).—The results given in the previous paper (B., 1925, 569) are extended for isochromatic and blue-sensitive plates, and correspond with those found for panchromatic plates. When the density is low p depends only on the exposure time. The value of p was not affected by wave-length as was found in the case of panchromatic plates. A theory of the deviations from the reciprocity law is discussed. In this connexion it is stated that it need only be considered that the deposit formed on full development is a measure of the action which has taken place. For the purposes of the theory, quanta are treated as molecules, obeying the law of mass action, and it is shown that two quanta are concerned in the photographic action, the two being absorbed within a short interval of time, giving the effect of two distinct absorptions when the exposure is short, and of a simultaneous absorption when the exposure is long. The two quanta must be taken up by different components of the absorber, and the maximum wave-lengths effective in the two stages must be close to the maximum for any photographic action. Either component may absorb the first quantum. The following formula is developed: $y = n(1 - e^{-2k^2It/k})$, where y is the number of molecules of the latent image, n is the number of double absorbers, and h and k are constants. It is considered that a single molecule of latent image is sufficient to make a grain developable. The formula is compared with results of Slade and Higson (B., 1921, 27 A). The assumption of one double absorber in a grain gives good agreement with the observed values, whilst the assumption of a large number in each grain is untenable. The fact that in the case of short exposures to intense light p rises above unity is ascribed to reversal, the theory of which is examined. It is assumed that reversal involves the absorption of four quanta. To explain certain discrepancies between theory and the observed results in the case of process plates, it is assumed that the two components of the double absorber are not exactly similar, and an equation is evolved which gives good results in the case of process plates. A similar formula can be developed on the assumption that there are two different kinds of absorber present, having similar components. W. CLARK.

Comparative study of a glycine and a metol–quinol developer. L. LOBEL and J. LEFÈVRE (Bull. Soc. Franç. Phot., 1927, **14**, 5—9).—Measurements show that the stated difference between the results of development in glycine and in metol–quinol is only an apparent difference, the two developers giving identical results as far as the final result is concerned. A glycine developer in 15 and 20 min. gave characteristic curves identical with those obtained with a metol–quinol formula in 2 and 4 min. By suitable dilution of the metol–quinol solution, the two developers gave practically identical results in equal development times. Identity of action was attained if a little potassium bromide was added to the metol-quinol to depress fog. W. CLARK.

PATENTS.

Colour photography or kinematography. L. DUFAY, and SOC. ANON. COMP. D'EXPLOIT. DES PROC. DE PHOT. EN COULEURS L. DUFAY (E.P. 262,386, 28.7.26. Conv., 4.12.25).—A selecting screen for plates or films for colour photography consists of two contiguous and separable multi-colour screen layers of different relative intensities of coloration, arranged so that similar coloured parts of each layer coincide exactly. The screen layers consist of a thin sheet of celluloid coated with a layer of transparent gelatin, which is dyed with dyestuffs having an affinity for both the gelatin and the celluloid, so that the celluloid is dyed to any desired intensity after the gelatin has been dyed throughout its thickness to any other desired intensity. The other side of the celluloid is coated with emulsion. The face of the celluloid is prepared to receive the gelatin coating by treating with ether, ethyl acetate, or acetone, so as to regulate the intensity of coloration of the celluloid. A substance such as baryta may be introduced into the emulsion so as to render it reflective. After the photograph has been taken, the gelatin selecting screen is washed off. W. CLARK.

Development of photographic plates, films, and the like. SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (E.P. 268,155, 22.4.26).—2 : 4-Diaminodiphenylamine is used as a developer for photographic plates etc. either alone or in conjunction with other developers such as metol and amidol, the usual methods of application being employed. A. J. HALL.

Production of multi-coloured bromoil transfer impression prints. F. SCHMID (E.P. 257,940, 2.9.26. Conv., 2.9.25).—A print is made in black from a bromoil matrix in the known way, and is then painted over with a water colour or tempora colour, dried, and then coated with a transparent resin varnish. The varnish is dried and a further print is then made, or else a number of prints alternating with coatings of varnish. A suitable varnish consists of an alcoholic solution of shellac, sandarac, and the lac known as "Zapon." W. CLARK.

Reversal process for direct positives. ICA A.-G. (G.P. 437,598, 26.2.24).—The developed silver image is treated with a mixture of lead nitrate and potassium ferricyanide, and the second exposure is given before the second development. The process can be used for paper, glass, or film images, and also for line and half-tone exposures. W. CLARK.

Process for obtaining direct positives by reversal. G. E. ZELGER, Assr. to PATHÉ CINÉMA, ANC. ÉTABL. PATHÉ FRÈRES (U.S.P. 1,628,445, 10.5.27. Appl., 14.4.23. Conv., 16.2.23).—See E.P. 211,431; B., 1924, 579.

Process of preparing sensitive layer on a suitable base. M. P. SCHMIDT and W. KRIEGER, Assrs. to KALLE & Co., A.-G. (U.S.P. 1,628,279, 10.5.27. Appl., 19.6.25. Conv., 28.5.24).—See E.P. 234,818; B., 1925, 692.

XXIII.—SANITATION; WATER PURIFICATION.

Waters with a solvent action on iron and manganese, the rôle of carbonic acid in metallic corrosion, and the determination of free carbonic acid in water. H. LÜHRIG (Gas- u. Wasserfach, 1927, **70**, 381—389).—The mechanism of the corrosive action of natural waters on metals is discussed and illustrated by examples from the author's practice. All waters exert a solvent action on iron or manganese in virtue of their hydrogen-ion content. In general, the free carbonic acid, determined by titration or by its solvent action on marble, gives a practical measure of the corrosive action of a water on metals, but exceptional cases may arise. Hard waters containing calcium bicarbonate can exert a solvent action on iron owing to the ease with which the bicarbonate decomposes giving free carbonic acid, although here the deposition of a layer of calcium carbonate on the metal may act as a protective layer against further attack. Water samples containing free carbonic acid readily lose carbon dioxide unless they are kept in bottles completely filled. In determining free carbonic acid by the action of the water on marble the usual time of contact can be very considerably shortened by shaking and then filtering before determining the hardness. A convenient and rapid method of determining free carbonic acid is based on its removal by the passage of a current of carbon dioxide-free air through the water, and titrating before and after. The method fails for waters with a high temporary hardness, but the corrosive action of such waters is of little importance. If necessary, however, they could be diluted with carbonic acid-free water before making the determination.
A. B. MANNING.

PATENTS.

Treatment of water. J. H. HARRINGTON, Assr. to G. W. ROBB (U.S.P. 1,623,342, 5.4.27. Appl., 2.2.24).—An apparatus for determining the chlorine content of treated water consists of three adjacent troughs, through which the water is passed in parallel, and in one of which it is treated with potassium iodide and starch. The colour so produced is compared with a maximum and minimum depth of colour formed in the other troughs by means of blue glass plates.
T. S. WHEELER.

Distilling apparatus. Water still. E. V. OLIVER (U.S.P. 1,625,222—3, 19.4.27. Appl., [A], 3.10.21. Renewed 6.10.24. [B], 4.12.23).—(A) A still comprises a casing which contains a well at the bottom, and a condenser and means for collecting condensate. A reservoir communicates with the well, and has a supply pipe brought in from above with a float valve; it has an open top, but has a lid of hinged sections meeting round the supply pipe with a fluid-tight flanged joint. (B) A condenser comprises vertical chambers alternately open at the top for cooling fluid and at the bottom for condensate. The cooling fluid is supplied by a many-branched pipe to the bottoms of the cooling fluids chambers. B. M. VENABLES.

Automatic [base exchange] water softener. C. P. EISENHAUER, Assr. to DURO Co. (U.S.P. 1,625,198, 19.4.27. Appl., 12.10.25).—A water softener comprising containers for softening material and for regenerating material is arranged so that the amount of water flowing through makes or breaks an electrical circuit which controls a needle valve that allows water pressure to reach hydraulically-operated valves which effect the change from softening to regeneration and back again.
B. M. VENABLES.

Water-softening apparatus. K. W. BARTLETT (U.S.P. 1,624,382, 12.4.27. Appl., 7.6.20).—The water passes through a heater to a settling tank of two chambers connected at their lower ends, from the second of which it passes through a valve to a filter, and through another valve to the service pipe. A connexion, with pump and valve, is also provided from the filter back to the first settling chamber, to which chamber the reagent is also added. B. M. VENABLES.

Apparatus for distributing water to be purified at different depths in the clarifier. BAMAG-MEGUIN A.-G. (G.P. 437,043, 17.3.25).—The apparatus conveying the water into the clarifier consists of two perpendicular cylinders, one fitting tightly inside the other, provided with sets of openings so arranged that by rotating one of the cylinders those at a desired depth are brought opposite to one another, thus admitting the water at that depth. L. A. COLES.

Apparatus for de-aerating water. J. SIM (Can.P. 261,617, 22.5.25).—The apparatus comprises a series of compartments each provided with an inlet and outlet, the last compartment being fitted with means for removing air and water vapour. Each inlet is provided with a valve controlled by a float inside the compartment it serves. L. A. COLES.

Water purifier in which the water is heated with steam and then filtered. ATLAS-WERKE A.-G. (G.P. 436,882, 11.7.25).—Continuous operation of the plant is rendered possible without the provision of an intermediate storage tank, by using water which has already been purified for injecting the fresh water into the apparatus. L. A. COLES.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 8, 1927.

I.—GENERAL; PLANT; MACHINERY.

Mathematical theory of the Michell ball viscosimeter: its design, construction, and operation. R. O. Boswall (Phil. Mag., 1927, [vii], 3, 994—1006).—Mathematical consideration of the Michell viscosimeter shows that it can only be applied in practice to measure viscosities over a range of temperatures with any degree of accuracy if the following conditions are fulfilled: (1) In order that there should be the least possible change of constant with change of radius, the projections used for regulating the initial film thickness at the centre of the cup must be correctly spaced to suit the initial difference in radius and height of projection. (2) To avoid unequal expansion, both cup and ball should be made of similar material. (3) The instrument should be calibrated over a range of temperature. (4) The use of a high-constant instrument with a very viscous fluid should be avoided. (5) When taking observations over a range of temperatures the rate of cooling should be slow. The depth to which the instrument is immersed in its bath does not affect its accuracy, provided that the edge of the cup is completely covered.

A. E. Mitchell.

Ewing ball-and-tube flow-meter. J. H. Awbery and E. Griffiths (Proc. Roy. Soc. Edinburgh, 1926, **47**, 1—10).—Experiments confirming and supplementing those of Ewing (*ibid.*, 1925, **45**, 308) are described. The steady and chattering modes of motion of the ball were observed, and the action of the meter is analysed. Calibration of a meter with a number of liquids showed that the rate of flow for any one position of the ball varies greatly from liquid to liquid. The influence of the angle of inclination of the tube was studied. The results are discussed by applying the theory of similitude. In general, it is not possible to deduce the calibration for one liquid by means of that for another.

W. Clark.

Patents.

Melting substances in jacketed vessels for carrying out chemical reactions. E. Legeler (G.P. 436,367, 7.7.25).—The space between the reaction vessel and the jacket is so subdivided that the whole or any portion can be heated according to the quantity of material to be melted or the amount of heat available. Alternatively, two or more concentrically-arranged heating cylinders may be employed, all of which are double-walled and subdivided as above.

A. R. Powell.

Atomising solid material. T. Goldschmidt A.-G., and V. Kohlschütter (G.P. 438,221, 5.8.19).—A mixture of the material in the form of vapour with a large volume of a gas is converted into a mist, and the particles are then precipitated electrically. Chemical change may take place during mist formation, or other constituents can be added to the mist, such as inert material which serves as a nucleus for the formation of the solid particles.

L. A. Coles.

Evaporation of solutions by spraying and subsequently drying the dissolved substance. O. Nordström (G.P. 437,257, 24.12.22).—The liquid is concentrated in an evaporator or other apparatus, and then sprayed into a long tube through which hot furnace gases are passed. At the end of the tube is a shaft packed with granular filtering material which retains and dries the solid recovered from the liquid used, whilst allowing the gases and vapours to escape. Any liquid which collects in the drying tube is run into a reservoir, whence it is pumped back through the spraying device into the hot end of the tube. The supply of liquid to the drying tube is regulated by means of floating valves in the feed tanks. In case the liquid contains dissolved or suspended combustible matter, the packing of the drying shaft consists of a granular fuel which is thereby enriched in combustible matter.

A. R. Powell.

Separation of solids from liquids. F. Bachmann, Assr. to Dorr Co. (U.S.P. 1,627,550, 10.5.27. Appl., 28.3.23).—A rotary screen is provided with means, spaced from it, for effecting a sweeping action of a current below the surface of the liquid to prevent accumulation of solids on the screen.

H. Holmes.

Separation of solid and liquid suspensions from air, gases, and vapours. P. Graefe (G.P. 436,366, 3.2.24).—The gases are drawn by centrifugal force or forced by means of a pump against the openings or conducting surfaces of a rotating system of double plates in such a way that the impurities are driven out between each pair of plates by centrifugal force into a collecting chamber. The apparatus consists of a system of solid plates, between each pair of which are others with perforated walls provided with openings and undulating surfaces so as to form hollow spaces through which the impurities are ejected whilst the gases pass through the perforations.

A. R. Powell.

Separation of soluble substances. Aktieselskapet Krystal (F.P. 612,993, 8.1.26. Conv., 12.1.25).—The leaching solution is passed through a column charged with the substance to be leached (*e.g.*, rock salt), thence to an evaporator where it is concentrated until supersaturated with respect to one substance (*e.g.*, sodium chloride). The hot solution is then passed through a column containing coarse crystals of the substance with which it is supersaturated whereby the excess

salt is deposited. The mother-liquor is diluted with condensed steam from the evaporator, and used again for leaching. A. R. POWELL.

Evaporator. J. PRICE, Assr. to GRISCOM-RUSSELL Co. (U.S.P. 1,617,081, 8.2.27. Appl., 12.6.23).—An evaporator for producing vapour from a scale-forming liquid consists of a shell for containing the liquid together with a number of easily removable steam-heating elements. These are slightly bent tubes which, under changes of temperature, exfoliate a substantial portion of the scale. S. PEXTON.

Distillation columns. DAMPFKESSEL U. GASOMETERFABR. A.-G. VORM. A. WILKE & Co. (G.P. 437,258, 2.7.25). —A distillation plant comprises a series of columns of different heights grouped together, and provided with separate inlet and outlet pipes and valves for the heating medium, the whole being surrounded by a heating jacket. A. R. POWELL.

Condenser for stills. E. V. OLIVER (U.S.P. 1,628,736—7, 17.5.27. Appl., 5.3.25).—The condenser casing is provided with bottom inlet passages for admission of the vapours and with spaced condensing chambers supported over openings in a horizontal partition. (A) The condensing chambers are separated by spaces, open at the top, receiving cooling water through pipes, from a reservoir closing the top of the casing, and an overflow from the casing is provided above the open tops of the cooling-water spaces. (B) The cooling water is supplied to a trunk pipe with branches passing through the wall of the casing and discharging close to the lower ends of the condensing chambers. H. HOLMES.

Separation or recovery of gases and vapours by solid absorbents. A. GODEL (E.P. 270,099, 28.5.26).—When regenerating solid absorbents by superheated steam, the issuing steam is superheated and/or compressed and used again to treat the same or another mass of absorbent, or, if the quantity of valuable constituents is large enough to be worth separating, the steam is condensed in an evaporator and the resulting secondary quantity of steam compressed and/or superheated for use. B. M. VENABLES.

Apparatus for extracting gases from liquids. MABAG, MASCHINEN- U. APPARATEBAU A.-G. (G.P. 436,858, 26.9.25).—A perforated drum situated above the liquid outlet, and serving to separate the liquid from the gases, encloses the float regulating the liquid and gas outlets. The device can be used in meters for measuring the volume of liquids. L. A. COLES.

Treating liquids with gases or vapours. J. RENNOTTE (F.P. 613,123, 15.7.25).—The gas or vapour is passed through a narrow tube into the bottom of every one of a series of superimposed closed tanks in such a way that a mixture of gas and liquid is forced upwards, on the injector principle, through larger pipes into the next higher tank, and siphons are arranged so that the liquid in all the tanks remains at the same height. A. R. POWELL.

Treating liquids or mixtures of liquids and solids with gases. A. BARTH (G.P. 437,483, 13.10.25). —The upper part of a reaction tower of the type described in G.P. 402,509 (B., 1925, 58) is connected by means of a U-tube, having the horizontal portion of the same diameter as the tower, with the upper part of a second similar tower. If desired, a series of these towers may be similarly joined together. The apparatus may be used for the manufacture of copper oxychloride and similar heavy-metal compounds. A. R. POWELL.

Regenerative furnace. A. W. SODERBERG (U.S.P. 1,629,056, 17.5.27. Appl., 10.6.21).—An open-hearth furnace is provided with a combined gas and air port and with an air port at each end of the hearth. Slag pockets arranged below each air port are connected to the ports by vertical flues. Butterfly valves mounted in these flues are operated simultaneously through lever connexions from hydraulic cylinders. H. HOLMES.

Control and reversal of regenerative furnaces. A. E. WHITE. From MORGAN CONSTRUCTION Co. (E.P. 270,184, 7.12.26).—A furnace hearth is provided at each end with an inlet–outlet, regenerator, and chimney stack, and in each stack is a jet of pressure air blown from individually regulable separate fans, and dampers are provided beyond the jets. In the outlet the fan is run fast with damper open, and the high-velocity air acts as an ejector, withdrawing the products of combustion from the furnace. In the inlet the damper is closed and the fan run slowly so that the air delivered strikes the damper, doubles back to the furnace, and is used for combustion. The dampers and fan regulators are interconnected so that reversal may be effected by the operation of one lever. B. M. VENABLES.

Calcining or other operations in rotary kilns. T. RIGBY (E.P. 270,356, 3. and 13.11.25).—Slurry other than cement slurry is atomised and delivered from separate sprays situated outside the kiln along the length of a stationary portion of the kiln, which is inclined towards the rotating part of the kiln. The supply of atomised slurry and of hot gases to the stationary part of the kiln is regulated so that there is no tendency for the dried slurry to cohere in large masses on the walls of the kiln (cf. E.P. 243,410 and 261,814; B., 1926, 129; 1927, 76). B. W. CLARKE.

Pulverising mill. O. SMITH and J. W. HAYES (E.P. 270,159, 18.10.26).—A disintegrator for easily friable substances such as grain is formed with a serrated lining and tangential inlet in the upper half of the casing and perforated screens in the lower half, and the shaft carries a number of saw-toothed discs, with the teeth set alternately to either side and centrally, and, if desired, beater blades between the discs. By means of a hood air may be caused to circulate out through the screens and back into the mill through an aperture in the side. B. M. VENABLES.

Grinding viscous materials. H. F. WINSLOW (U.S.P. 1,628,211, 10.5.27. Appl., 29.6.21).—The material is passed over a sequence of grinding rolls. That near the ends of a later roll is returned to an earlier roll, and the remainder delivered from the later roll constitutes the finished product. H. HOLMES.

Device for compressing viscous materials into high-pressure vessels. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES (F.P. 606,190, 12.2.25).—The material is propelled by a screw through a check-valve into a compression chamber, and thence by a horizontally

moving piston through a second valve into the high-pressure vessel. The device is used in the production of hydrogen from iron and water under high pressure, and in the pressure oxidation of paraffins.

A. B. Manning.

Centrifugal machine. E. Roberts, Assr. to Western States Machine Co. (U.S.P. 1,627,868, 10.5.27. Appl., 24.12.21).—A non-rotary spindle is supported from a hanger to gyrate upon a spherical ball-joint which prevents axial movement of the spindle in either direction, passages being provided in the spindle for conveying lubricant to the joint. The basket shaft is mounted to rotate upon and to swing with the spindle.

H. Holmes.

Filter. E. Robertson (U.S.P. 1,629,085, 17.5.27. Appl., 21.9.23).—An upright cylinder is provided with passages extending outwards from its top and bottom. Filtering material of progressively finer texture from bottom to top rests on the bottom around the lower passage with its top layer spaced from the upper passage. Inlet and outlet means are provided, each with valved connexions to the upper and lower passages. The upper passage is connected to a combined filtering-outlet and washing-inlet nozzle, spaced from the filtering material and provided with a lower opening directed downwards and with various lateral openings. H. Holmes.

Pulp thickeners or filters. R. Haddan. From Oliver Continuous Filter Co. (E.P. 270,108, 11.6.26).—A number of tubular filter elements are continually and entirely submerged in a tank of the pulp to be thickened, and their interiors are subjected alternately to vacuum and pressure, preferably in groups, so that there is always some filtrate being withdrawn, the valve for the purpose being placed below the liquid level in the tank. Means are provided to cause the thickened mud to travel continuously to a central outlet, and within the filter tube a short length of unperforated tube depends, which acts as a siphon trap to provide liquor, which, on application of the positive pressure, is blown as a back-wash through the filter before the air can get through.

B. M. Venables.

Separating out from gas mixtures the constituents that are readily condensable. Ges. für Linde's Eismaschinen A.-G. (E.P. 258,856, 9.9.26. Conv., 25.9.25).—The liquefiable constituents are separated by cooling the gases by expansion. A preliminary cooling of the gases after compression is made in an evaporator cooler through which flow the expanded gases from which the liquefiable constituents have been removed. The expanded gases are sprayed with water in the cooler, and by evaporation of the water the compressed gas is cooled to 0°, and any water condensed separated.

A. C. Monkhouse.

Continuous absorption by porous material of constituents of gas mixtures. I. G. Farbenind. A.-G., Assees. of W. Simmat (G.P. 438,176, 15.10.22).—Absorbent material, such as activated charcoal, glides slowly down a spiral tower, and the gases pass up the tower.

L. A. Coles.

Continuous analysis of gas currents by absorption of the absorbable constituents. Wega m.b.H. Wärmetechn. Ges. Aachen (G.P. 437,563, 11.11.24).—A portion of the gas is passed into a lens- or prism-shaped measuring vessel connected at its lowest point through a U-tube with a vessel containing the absorbing medium in such a way that the reduction of pressure produced by absorption of one of the constituents of the gas causes the absorbing medium to be drawn into the measuring vessel. A. R. Powell.

Filtering arrangement for a flue-gas recorder. Siemens & Halske A.-G., Assees. of H. Grüss (G.P. 437,936, 10.2.25).—The filter is arranged in a gas-tight chamber in the wall of the setting or flue; an inlet pipe leads to the chamber from the point at which the gas to be tested is to be taken. A position where the wall temperature lies between 200° and 400° is suitable for the chamber. A. B. Manning.

Carrying out chemical reactions or physical processes. E. Buhtz (U.S.P. 1,629,200, 17.5.27. Appl., 6.4.25. Conv., 7.2.25).—See E.P. 250,722; B., 1926, 520.

Crushing or grinding machine. A. C. Hamey (E.P. 247,943, 10.2.26. Conv., 19.2.25).—See U.S.P. 1,627,506; B., 1927, 431.

Refrigeration. B. C. von Platen and C. G. Munters, Assrs. to Electrolux Servel Corp. (U.S.P. 1,629,733, 24.5.27. Appl., 7.12.26. Conv., 18.8.22).—See E.P. 202,602; B., 1924, 321.

Sterilising and preserving liquids. R. Seligman (E.P. 270,030, 17.2. and 8.3.26. Addn. to E.P. 229,435).

Separating the solid and liquid constituents of a material (E.P. 270,066).—See II.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Dehydration of alcohol [for motor spirit]. K. Petrlik (Bull. Assoc. Chim. Sucr., 1927, 44, 223—224).—The air required for the combustion of a mixture of 95% alcohol and petrol in equal parts contains on the average about 2·5 times as much water as there is in the alcohol. Consequently there is no advantage in using absolute alcohol in making mixed motor spirits, especially as, by the addition of 10% of benzene to the 1 : 1 alcohol-petrol mixture, no separation of the constituents takes place above 35°. A. R. Powell.

Titrimetric determination of hydrogen sulphide in producer gas. I. Ciochină (Bul. Soc. Chim. România, 1926, 8, 126—127).—A measured quantity of the filtered gas is passed into calcium hydroxide solution; the hydrogen sulphide absorbed is swept out by a current of carbon dioxide into two vessels containing (*a*) 100 c.c. of a solution made by dissolving 28 g. of cadmium acetate, 112 g. of zinc acetate, and 280 g. of sodium acetate in 7 litres of water containing 5 c.c. of acetic acid, and (*b*) 50 c.c. of this solution diluted with 50 c.c. of water. Zinc and cadmium sulphides are precipitated; standard iodine solution is added, followed by 25 c.c. of hydrochloric acid (1 : 3), and the mixture titrated with standard sodium thiosulphate solution. S. K. Tweedy.

Simple analysis of crude Rumanian petroleums

and of the petroleum fractions separated by filtration. G. GANE and (MME.) A. METTA (Bul. Soc. Chim. România, 1926, 8, 100—115).—After removal of the fraction volatile below 100°, the carbon and nitrogen were determined by Pregl's method and the sulphur by combustion in a Berthelot bomb. Analysis of the different fractions indicates that an abnormal concentration of sulphur tends to occur in the fraction of b.p. 150—180°. This may be due to the formation of a trimethylthiophen (cf. Challenger and others; B., 1926, 617). The results show that, for equal density, the carbon content of Rumanian petroleums is higher than that of American oils, partly because of the neglect of the very volatile fraction in the analyses and partly because of the higher proportion of naphthalenic and benzenoid hydrocarbons. The nitrogen content is practically constant at 0·3%, in marked contrast to American oils. The average sulphur content is 0·23%, and varies only slightly. Preliminary results of analyses of petroleum fractions separated by adsorption in siliceous earth are recorded. S. K. TWEEDY.

Bleaching action of fuller's earth. NEUMANN and KOBER, also KEPPELER.—See XII.

PATENTS.

Production of coke briquettes with inorganic binding agents. L. WEBER (E.P. 269,655, 20.1.26. Addn. to E.P. 243,129; B., 1926, 42).—An inorganic substance which evolves a gas on being burnt may be used to briquette low-temperature coke. Such are sulphite-liquor, and the solid product derived from it ("cell pitch"). Materials, *e.g.*, clay and cement, giving little or no gas may be used in conjunction with those having gas-forming properties. R. A. A. TAYLOR.

Briquetting coal and coke. E. KLEINSCHMIDT (G.P. 433,237, 7.5.24).—A stream of finely-ground pitch, asphalt, resin, etc., or a dispersed spray of tar is blown simultaneously with a binder into a stream of finely-ground coal or coke to be briquetted. The process requires only the smallest quantity of binder. S. PEXTON.

Briquetting coal. G. MÜLLER and J. FISCHER (F.P. 611,548, 22.2.26).—The coal is mixed with a small quantity of a combustible binder such as the protein or latex of plants, *e.g.*, rubber latex, and briquetted by a known process. The rubber latex can be used in solution in a volatile liquid, and the briquetting then so conducted that the solvent is wholly or partly vaporised. A. B. MANNING.

Draining and concentrating coal and other slimes. SIMON-CARVES, LTD., and A. ROBINSON (E.P. 269,967, 25.1.27).—The slurry separated by settling in the main tank of the washery is passed over a vibrating sieve. The liquid passing through the sieves is returned for circulation in the washery after removal of the useless mineral matter in it by gravity separation. The liquid from the slurry may be settled in a tank having a tapered or sloping bottom, or a number of conical depressions, or one or more weirs. Further, the slurry may be sieved over a pair of independent vibrating sieves. A substantially horizontal, rapidly vibrating sieve may be used. R. A. A. TAYLOR.

Treatment of coal [to prevent smoke formation]. J. F. LAHART, J. S. LAHART, and H. A. PIERCE (F.P. 610,879, 12.2.26).—By mixing coal with 1·1—2·2% of a mixture of lime, salt, and Portland cement the fuel value is raised and smoke formation is prevented. A. B. MANNING.

Treatment of peat. ALLGEM. KOMMERZGES. A.-G., and A. MANNESMANN (E.P. 269,993, 1.2.26).—The peat is treated in a centrifugal device and, as the material is thrown outwards, its cells are burst so that the water is separated; rigid obstructions may be placed in its path out of the centrifuge to break it up, or a guide is placed just outside the disc of the centrifuge so that as the peat is thrown outwards a grinding action may take place between the guide and the edge of the disc. Freed from its water, the peat is allowed to remain until it has formed a solid cake of lignitic or coal-like appearance of *d* 1·23; the consolidation may be accelerated by heat, and the product pressed without a binder, but preferably with heating, into briquettes. The delivery of the peat substance to the moulding device may be continuous, and pieces the size of the briquettes may be cut off. The crude peat is pressed into as thin a layer as possible for delivery to the centrifuge, from which the atomised water is removed by suction. R. A. A. TAYLOR.

Separating the solid and liquid constituents of a material [peat] from one another. ALLGEM. KOMMERZGES. A.-G., and A. MANNESMANN (E.P. 270,066, 30.3.26. Addn. to E.P. 269,993; cf. preceding abstract).—Material such as peat or lignite is fed to the centre of a rapidly-rotating concave disc or saucer provided with pins; the material is broken up by the pins, and the various solid constituents are flung off to various distances and fall into separate compartments, whilst the liquid constituents are atomised. There may be several co-axial discs of increasing diameter rotating in the same or opposite directions. B. M. VENABLES.

Drying fuels in stages by internal heating in a shaft-drier. METALLBANK & METALLURGISCHE GES. A.-G. (G.P. 437,099, 24.6.23).—The drying medium, which is circulated through the apparatus, is heated before each stage of the drying process by the admixture of hot gases, which are supplied to gas-mixing chambers built into the shaft-drier itself. A. B. MANNING.

Recovery of the drying agent retained by moist fuels after being dried. E. BERL (G.P. 435,667, 20.2.25. Addn. to G.P. 419,906; B., 1926, 308).—The drying agent retained by fuels which have been submitted to the process described in the main patent is recovered by passing superheated steam through the material, which is preferably externally heated also, and cooling the mixed vapours to a temperature just above the condensation temperature of the steam, thereby condensing the greater part of the drying agent. The steam is again superheated and used for the recovery of a further quantity of the drying agent, either at atmospheric or other pressures. A. B. MANNING.

Production of carbon black. C. MATLOCK, Assr. to MONROE-LOUISIANA CARBON CO. (U.S.P. 1,617,071—2 and 1,617,074, 8.2.27. Appl., [A], 2.6.21, [B], 21.7.21.

Renewed 30.10.26, [c], 16.11.21).—(a) Liquid hydrocarbons are partially burnt, and the carbon black which is formed is separated from the products of combustion at a temperature above 425°. (b) Hydrocarbons are partially oxidised and the carbon black produced is collected on a screen which is continuously being passed through the products of combustion. The carbon is scraped from the screen after it has passed from the furnace flue. The carbon is thus separated from the combustion products at a temperature above the ignition point of the hydrocarbon from which it is formed. (c) A suitable furnace and burner for the production of carbon black by the incomplete combustion of hydrocarbons are described. S. Pexton.

Manufacture of absorbent carbon. E. Urbain (E.P. 269,961, 20.1.26).—A pulverised charcoal is formed into a plastic mass with a suitable binding material. This is extruded before calcination, or it may be moulded. In order to obtain a product of different absorptive properties, the amount of water in the agglomerant, or of the pressure of extruding or moulding, or both, is varied. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 218,242 (B., 1925, 435), and in pursuance of Sect. 8, Sub-sect. 2, to E.P. 247,241 (B., 1926, 308).] R. A. A. Taylor.

Production of highly active contact material. I. G. Farbenind. A.-G., Assees. of O. Nicodemus (G.P. 438,071, 14.6.24).—Carbonisable material with a cellular structure, such as peat or wood, is saturated with catalytic material in solution or in the colloidal form, and, after compression in moulds, is carbonised in the presence of gases containing ammonia, and then calcined. L. A. Coles.

Reactivation of charcoal. Algem. Norit Maatschappij (G.P. 433,523, 1.7.25).—The charcoal is reactivated by treatment with an acid solution under pressure. S. Pexton.

Production of [layers of] especially hard carbon [upon other material]. Siemens & Halske A.-G., Assees. of C. A. Hartmann (G.P. 438,429, 5.5.25).—Material such as quartz, porcelain, or charcoal is coated with hard carbon by exposing it while hot to the action of a gaseous carbon compound at a lower temperature, in a vessel from which the air has been exhausted or replaced by a non-reacting gas, such as nitrogen or argon, the volume of the gaseous carbon compound being small compared with that of the containing vessel. L. A. Coles.

Production of carbon dioxide for fertilising plants, by burning coal. F. Hörning (G.P. 438,186, 15.7.25).—Combustion gases drawn from the top of a furnace provided with a secondary air supply to prevent formation of carbon monoxide flow downwards together with a current of water through a supply of purifying material, and thence to the place of use. L. A. Coles.

Motor fuels. I. G. Farbenind. A.-G., Assees. of M. Bachstez (G.P. 437,276, 24.7.25).—"Knocking" is prevented by the addition of alkyl substitution products of silicon hydride, *e.g.*, silicon tetraethyl, to fuels for internal-combustion engines. A. B. Manning.

Motor fuel. N. Blank (F.P. 612,227, 10.3.25. Conv., 11.3.24).—A hydrocarbon, *e.g.*, petroleum or cleaning oil, is treated with fuming nitric or concentrated sulphuric acid and a deoxidising agent (lime, chalk, etc.), and the product mixed with benzine or benzene. The motor fuel so prepared should be free from unpleasant odour, and should produce no smoke. A. B. Manning.

Motor fuel mixtures. A.-G. für Petroleumind., and M. Herrmann (G.P. 437,322, 21.7.22).—Aldehydes, *e.g.*, acetaldehyde, are added to motor fuels containing alcohol, petroleum, and benzine or benzene, or both, in order to render the mixture homogeneous, the addition lowering considerably the temperature of separation. A. B. Manning.

Gasification of raw fuel in a multiple chambered producer. F. Jahns (G.P. 398,542, 25.2.21).—The producer is designed to yield gas of low moisture content. Gas from the chambers in the early stage of gasification is introduced into the chambers where the final stages of gasification are proceeding. S. Pexton.

Removal of benzol from gases containing the same. I. G. Farbenind. A.-G. (E.P. 257,906, 10.8.26. Conv., 1.9.25).—The gases from which the benzol is to be removed are washed with the products, or fractions of the products, obtained by the destructive hydrogenation of carbonaceous materials. R. A. A. Taylor.

Apparatus for separating particles from gases at high temperatures. C. Matlock, Assr. to Monroe-Louisiana Carbon Co. (U.S.P. 1,617,073, 8.2.27. Appl., 16.11.21).—Hot flue gases containing carbon particles are filtered through vertical tube screens which are supported from the top of a chamber by rods and springs. The lower ends of the screens engage in holes in a header plate. Gas passes through the screens from the inside, and the carbon which is deposited is brushed from the tubes into a receiving hopper attached to the bottom of the chamber. S. Pexton.

Apparatus for purifying gases by filtration through loose solid material. B. Christoffels (G.P. 438,269, 15.1.24).—Channels for distributing the gases through the material lie in a plane parallel to that of the surface of the filter medium. L. A. Coles.

Gas producers. G. H. Bentley and E. G. Appleby (E.P. 269,689, 26.1.26).—The air supplied to the generator is heated by being passed all round the wall of the generator, and then upon entering the generator it comes into contact with a spray of water. The water may also be preheated. An intimate mixture of air and water may be made. R. A. A. Taylor.

Gas producers [for wood]. J. R. F. M. Lasmolles (E.P. 262,088, 15.11.26. Conv., 27.11.25).—The producer consists of an upper furnace into which the wood is fed from a hopper. Air preheated in the dome of the furnace enters at the top and side of the furnace; a partial combustion of the wood takes place, and the charcoal formed falls through the grate into a lower furnace in which are a number of balls of refractory earth or porcelain. Air is admitted to this furnace through adjustable or automatic valves, the charcoal is gasified, and the gases produced from both furnaces are withdrawn

from below the grate of the lower furnace, and, after removal of dust, pass to the condensing system.
A. C. Monkhouse.

Manufacture of water-gas. W. J. Mellersh-Jackson. From Western Gas Construction Co. (E.P. 269,221, 8.10.25).—The apparatus consists of a generator, a superheater, and a carburettor. An air-blast may be passed in each direction through all of these, so that surplus heat is recovered from the superheater and restored to the fuel-bed, reconditioning it from above and below. Alternatively, the air-blast may be passed upward through the generator cold. The gas made may be recovered. Steam may be added to the air-blast. Various predetermined cycles may be adopted for running the plant.
R. A. A. Taylor.

Manufacture of gas from oil or tar (hydrocarbons) or for cracking oil or tar. K. N. Wannebo (E.P. 269,711, 16.3.26).—The cracking or gasifying operation is conducted in a heat-retaining generator. The oil or tar is admitted near the middle of the generator, and air for combustion for generating the heat necessary for the reaction is admitted alternately from opposite ends of the chamber. The air consumes mainly carbon and lampblack deposited during the preceding period of the process. The produced gases are taken off at the end opposite to that at which the air is admitted. After shutting the air intake, some steam may be admitted at the same end of the chamber as the air. Unconsumed combustion air is thus expelled before the process is reversed. Steam may also be admitted in small quantities intermittently or continuously throughout the gasifying period.
R. A. A. Taylor.

Drying of fuel gases. W. C. Holmes & Co., J. Parker, and D. M. Henshaw (E.P. 268,429, 31.12.25).—Fuel gases after a partial drying by hygroscopic liquids are further dried by passage over silica gel or over carbides, *e.g.*, calcium or aluminium carbide. A. C. Monkhouse.

Detecting, indicating, and recording the presence of inflammable vapours or gases. H. T. Ringrose (E.P. 267,990, 28.9.25 and 31.3.26).—The gas to be tested is aspirated or allowed to diffuse through a porous vessel containing a heated platinum wire, and the diminution in pressure due to the combustion of the gas is measured by a recorder. To correct for fluctuations of temperature, pressure, and humidity a second porous vessel is attached to the other limb of the recorder. This latter vessel either is protected from exposure to the inflammable vapours or has the heating element enclosed, or the temperature of the heating element is maintained below the ignition point of the vapours. A. C. Monkhouse.

Carbonisation of bituminous coal in a rotary retort. Gewerkschaft M. Stinnes, and A. Weindel (G.P. 437,813, 28.12.22).—The charging end of the retort is heated externally so that the temperature of the whole of the coal to be carbonised is raised at once to the point at which tar formation begins. A. B. Manning.

Carbonisation of combustibles at low temperatures. C. Pamart (E.P. 268,613, 15.7.26).—The material to be carbonised is fed by a conveyor into a rotating inclined drum which is heated externally at 500—600°. In the drum are a number of steel balls or discs to aid the transmission of heat and to prevent caking. A number of holes at the lower end of the drum act as a sieve, the balls and refuse are retained, and the pulverised semi-coke falls into an enclosed chamber from which it is withdrawn by a conveyor. The speed of rotation of the drum can be varied, and the gases produced from the carbonisation are withdrawn from the lower end of the drum. A. C. Monkhouse.

Continuous distillation of tar and oil. E. Blumner (G.P. 432,728, 6.5.25. Addn. to G.P. 340,991; B., 1922, 407 a).—The mouth of the tube for feeding tar or oil into a container of molten metal has a jet which distributes it as a fine spray uniformly over the whole section of molten metal. S. Pexton.

Distillation of carbonaceous substances. E. R. Sutcliffe (E.P. 268,080, 28.9.25 and 28.6.26).—The material to be carbonised is heated internally at a maximum temperature of 750° by preheated steam or steam and gas. In a particular example 100 lb. of steam are used per 100 lb. of coal, and the temperature of the gases leaving the retort is 150—175°. The gases produced are sprayed with heated water or liquor in a scrubber and also in the gas main. Any tar condensed is separated, and the hot liquor is re-circulated by a pump. A suitable high-pressure steam ejector is combined in the system for the removal of steam from the circulating liquor; the steam then passes to the regenerator and on to the retort. A. C. Monkhouse.

Vertical retort. J. Trautmann (G.P. 430,365, 6.10.22).—The heating walls are built up of hollow triangular-sectioned bricks superimposed at slight intervals. The inclined surfaces of the bricks form sliding surfaces for the charge, and the intervening spaces allow of the escape of the volatile products. S. Pexton.

Retort setting for the carbonisation of wood. Soc. Anon. des Anc. Établ. Loy et Aubé (G.P. 436,996, 19.11.24).—The retorts, which are of the horizontal, closed, and externally-heated type, are supported on rollers within the setting, from which they are removed for charging and discharging. They can be used also for quenching the charcoal. A. B. Manning.

Ovens for distilling coal. J. Daniels (E.P. 245,764, 1.1.26. Conv., 8.1.25. Addn. to E.P. 239,884; B., 1927, 133).—In a battery of ovens having vertical combustion flues, transverse flues are provided in the roof of the oven. The heating walls are divided into groups each with its own regenerator system, and these groups are connected in pairs by the transverse flues. Where each group has two regenerators each connected to a wall flue, the regenerators are used alternately either for preheating the air or one for heating the air and the other the diluent gas. Expansion joints are provided between the groups of heating walls.
A. C. Monkhouse.

Refining low-temperature benzines. Zeche M. Stinnes, Assee. of F. Müller and P. Hützen (G.P. 437,048, 18.10.23).—The crude product is washed with concentrated pure or denatured alcohol, separated, and, if necessary, distilled; or water is added to the alcohol-benzine mixture after washing, producing a smooth separation of the benzine and wash-alcohol. Washing losses are thereby reduced to a minimum.
A. B. Manning.

Production of activated carbonaceous substances. E. R. Sutcliffe (U.S.P. 1,629,237, 17.5.27. Appl., 17.1.20. Conv., 1.2.19).—See E.P. 166,202 ; B., 1921, 618 a.

Cracking or fixing the illuminating constituents of coal gas. Kohlenveredlung G.m.b.H. (E.P. 246,490, 21.1.26. Conv., 23.1.25).—See F.P. 606,886 ; B., 1927, 210.

Removal of sulphuretted hydrogen from gases. W. Gluud, Assr. to Koppers Co. (Re-issue 16,631, 7.3.27, of U.S.P. 1,597,964, 31.8.26).—See B., 1926, 908.

Apparatus for the determination of gases [carbon monoxide]. S. H. Katz (E.P. 249,088, 15.2.26. Conv., 12.3.25).—See U.S.P. 1,578,666 ; B., 1926, 430.

Process of cracking hydrocarbon oils. W. F. Faragher and W. A. Gruse, Assrs. to Gulf Refining Co. (U.S.P. 1,629,908, 24.5.27. Appl., 15.1.21).—See E.P. 174,085 ; B., 1923, 301 a.

Acetylene generator. A. Wagner (Swiss P. 116,964, 28.7.25).

Compressing viscous materials (F.P. 606,190). See I.

Bituminous concrete (E.P. 269,975).—See IX.

Medicinal waters [from coal] (E.P. 269,660).—See XX.

III.—TAR AND TAR PRODUCTS.

Nitrogen balance-sheet in the distillation of tar. H. ter Meulen (Rec. trav. chim., 1927, **46**, 284—286).—When tar is distilled, the nitrogen found in the volatile products and in the coke is less than that in the original tar ; the difference, which may be as great as 45%, has been generally attributed to the formation of free nitrogen. It is, however, now shown that the Kjeldahl method of determining nitrogen, whilst fairly accurate for the tar, gives much too low a value for the coke. Analysis by catalytic hydrogenation (A., 1925, ii, 599) gave 1·37—1·39% for the nitrogen in a sample of coke for which the Kjeldahl value was 1·03—1·04%. The balance of nitrogen lost in the distillation of tar, determining nitrogen in tar, volatile products, and coke by the new method, is now found to vary from 1·2 to 8% ; slow distillation causes only a slight increase in the loss. E. W. Wignall.

Patents.

Resolution of emulsions or suspensions containing tar or oil. H. W. Robinson and D. W. Parkes (E.P. 268,547, 30.3.26).—Finely-divided mineral powder such as granite or slate dust, which is more easily wetted by water than by oil, is added to an emulsion containing 30—35% of water, and the mixture heated at about 100°. The powder and water agglomerate, and the oil separates to the top. With emulsion containing more than 40% of water, the emulsion is added to the powder in the cold, when a preliminary separation of water occurs. If further emulsion be added and the mixture heated, a reversal of the phases takes place, and the oil separates out. The process is applicable to creosote and tar emulsions, and with a product mixed to contain 33% of water, 96% of oil was recovered using 0·7 lb. of powder per gallon. Emulsions which are strongly alkaline are rendered slightly acid before treatment. A. C. Monkhouse.

Distillation plant [for coal tar]. J. N. Hazeldon (E.P. 264,591, 21.10.25).—A series of shallow trays containing a heating element is built in the upper part of the still. Tar etc. to be distilled is fed on to the trays, and flows in contact with the heating element into the bottom of the still. The tar is thus dehydrated before it reaches the main body of the still, and priming is avoided. S. Pexton.

IV.—DYESTUFFS AND INTERMEDIATES.

Patents.

Production of azo dyestuffs containing chromium. H. Gubler, H. Stahel, and F. Straub, Assrs. to Soc. Chem. Ind. in Basle (U.S.P. 1,623,005, 29.3.27. Appl., 21.6.26. Conv., 3.7.25).—Unsulphonated azo dyes of the general formula R′N : NR″, where R′ is a benzene nucleus containing at least one hydroxyl group ortho to the azo bridge, and R″ is 1-phenyl-5-pyrazolone with the azo group in position 4, and at least one of R′ and R″ contains a sulphonamido-group, are treated with tervalent chromium compounds to yield acid *dyes*, which give fast, level, orange shades on animal fibres. *E.g.*, *o*-amino-*p*-sulphonamidophenol is nitrated to 2-*nitro*-6-*amino*-4-*sulphonamidophenol*, m.p. 191°, which is diazotised and coupled with 1-phenyl-3-methyl-5-pyrazolone to yield an acid *dye*, which gives on wool yellow-red shades, orange on after-chroming. T. S. Wheeler.

Manufacture of azo dyestuffs derived from 2-naphthol-3-carboxylarylamides. I. G. Farbenind. A.-G., Assees. of H. Wagner (U.S.P. 1,622,690, 29.3.27. Appl., 17.3.22. Cf. E.P. 210,462 ; B., 1924, 708).—Diazotised mononuclear aromatic amines are coupled with 2-naphthol-3-carboxylo-*o*-toluidides to give fast *dyes*, especially if one, and preferably both, components contain halogen. *E.g.*, material padded with 2-naphthol-3-carboxylo-*p*-chloro-*o*-toluidide, and then treated with *p*-nitro-*o*-anisidine, is dyed fast, bright red shades. T. S. Wheeler.

Production of azo dyes for dyeing cellulose esters, especially cellulose acetate. Chem. Works, formerly Sandoz (Chem. Fabr. vorm. Sandoz) (E.P. 245,758, 30.12.25. Conv., 6.1.25).—Azo dyes solubilised by the introduction of glycol or glycerol residues are used for dyeing cellulose acetate. Thus aminoaryl ethers of glycol or of glycerol may be diazotised and coupled with suitable azo components, carboxylic and sulphonic groups being absent. C. Hollins.

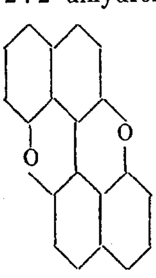
Manufacture of sulphurised derivatives of phenols and naphthols, and their application as mordants. Fabr. van Chem. Producten, and E. Kraus (E.P. 269,970, 26.1.26).—The sodium salt of a phenol or naphthol is heated with sulphur in the presence of a non-aqueous medium, which is preferably a further (limited) quantity of the phenolic compound. The products may be treated with formaldehyde or acetaldehyde and a sulphite. The initial products are used as mordants for basic dyes on cotton and for reserving textiles ; the aldehyde-sulphite treatment yields tanning agents.

Suitable proportions are 5 mols. of a phenol, 1 mol. of alkali (aqueous alkali may be added to the phenol and water removed by evaporation), 5—10 atoms of sulphur, $2\frac{1}{2}$—5 mols. each of aldehyde and sulphite. The first reaction takes 6—24 hrs. at 160—180°, or 50—60 hrs. under reflux in solvent naphtha at 150—160°. The product is a yellow resin, which is soluble in alkalis, and becomes acid-soluble by treatment with sodium sulphite and formaldehyde or acetaldehyde at 70—90° for $\frac{1}{2}$—2 hrs. In the examples phenol, commercial cresol, and β-naphthol are used. C. Hollins.

Manufacture of condensation products of the anthraquinone series. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 245,165, 28.12.25. Conv., 27.12.24. Addn. to E.P. 205,502; B., 1924, 627. Cf. also E.P. 222,125; B., 1926, 148).—Dibenzanthrone or 4 : 4′-dibenzanthronyl is heated with an aliphatic or aromatic acid chloride in the presence of aluminium chloride and oxygen or a halogen. Dibenzanthronyl gives in this way with benzoyl chloride a pure blue vat *dye*; with oxalyl chloride a greyish-blue vat *dye*; with carbonyl chloride a grey to black vat *dye*. If halogen be used in place of oxygen, halogenated products are obtained, some of which vat with difficulty. C. Hollins.

Preparation of leuco-hydroxyanthraquinones. I. G. Farbenind. A.-G., Assees. of K. Weinand (G.P. 436,526, 4.7.25).—Hot alkaline reduction (*e.g.*, with alkali and sodium hyposulphite) of hydroxylated anthraquinones containing α-amino-groups, or their β-substituted derivatives, replaces the α-amino-groups by hydroxyl and β-substituents (sulphonic groups) by hydrogen, giving leuco-hydroxyanthraquinones, which may be salted out with sodium sulphite. Thus, 1-amino-4-hydroxyanthraquinone, boiled for 1 hr. with sodium hydroxide and sodium hyposulphite, yields leuco-1 : 4-dihydroxyanthraquinone (leucoquinizarin). 4 : 8-Diaminoanthrarufin, 1-amino-4 : 5 : 8-trihydroxyanthraquinone, and 4 : 8-diaminoanthrarufin-2 : 6-disulphonic acid similarly give leuco-1 : 4 : 5 : 8-tetrahydroxyanthraquinone. C. Hollins.

Preparation of dinaphthylene dioxide [and vat dyes therefrom]. R. Thyll and W. Schmid (Swiss P. 114,913, 30.4.25).—The dried *copper* compound of 2 : 2′-dihydroxy-1 : 1′-dinaphthyl (di-β-naphthol), prepared by adding copper sulphate solution to a hot alkaline solution of the dinaphthol and making alkaline again with ammonia, is heated at 220—300° in a current of air or steam. Dinaphthylene dioxide, $C_{20}H_{10}O_2$, m.p. 240°, identical with the product obtained by Bünzly and Decker (A., 1905, i, 884) sublimes, and a further quantity may be obtained from the residue by removal of the dinaphthol with alkali. The solution of dinaphthylene dioxide in concentrated sulphuric acid yields when poured into water a mixture of a *sulphonic acid* and a hydroxylated *product*. A sulphonic acid is obtained by the action of hot sulphuric acid, and *nitro*- and *bromo*-compounds are described. Oxidation of dinaphthylene dioxide in acid solution gives a *quinone*, which on bromination yields a violet vat *dye*, and on nitration and reduction brown to blue vat *dyes* according to the degree of nitration. The *N*-alkyl and *N*-aryl derivatives of these aminoquinones are also vat dyes, and the N-*benzoyl* derivative of the polyaminoquinone is a bordeaux-red vat dye, very fast to chlorine. All these vat dyes show good light- and acid-fastness, and require only a weakly alkaline vat. C. Hollins.



Preparation of chlorinated perylenes and their derivatives. Comp. Nat. de Mat. Col. et Manuf. de Prod. Chim. du Nord Réunis (Établ. Kuhlman) (F.P. 611,017, 12.2.26).—Further to E.P. 244,739 (B., 1926, 974), perylene derivatives are chlorinated smoothly in nitrobenzene solution with nascent chlorine. *iso*Violanthrone and 3 : 10-perylenequinone give dichloro-derivatives. C. Hollins.

Preparation of 1 : 3 : 5-triazine-2 : 4 : 6-tricarboxylic chloride [and vat dyes therefrom]. E. Ott (Swiss P. 111,562, 20.9.24. Conv., 13.9.24).—Cyanurictricarboxylic esters are heated with phosphorus pentachloride. Ethyl 1 : 3 : 5-triazine-2 : 4 : 6-tricarboxylate and phosphorus pentachloride, heated at 130—140° until no more gas is evolved and the mass remains completely liquid when cold, yield on fractional distillation 1 : 3 : 5-*triazine*-2 : 4 : 6-*tricarboxyl chloride*, b.p. 150—155°/1 mm. This reacts with amines in nitrobenzene solution to form amides, those from aminoanthraquinones being valuable vat dyes. C. Hollins.

Manufacture of nuclear alkylated or *cyclo*alkylated arylsulphonic acids. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 250,241, 30.3.26. Conv., 1.4.25).—The method of E.P. 242,233 (B., 1927, 71) is improved by the use of chlorosulphonic acid in place of sulphuric acid, and is extended to other aromatic hydrocarbons besides naphthalene and to other alcohols (*e.g.*, butyl alcohol, *cyclo*hexanol, commercial fusel oil, etc.). Condensation and sulphonation may be combined in a single operation. The products are said to have enhanced wetting-out action probably as a result of the intensive sulphonation. The condensation of naphthalene in presence of chlorosulphonic acid with *n*-butyl alcohol at 80—100°, and with *cyclo*hexanol at 70°, is described, simultaneous sulphonation occurring in both cases. C. Hollins.

Manufacture of stable diazo compounds. G. de Montmollin and G. Bonhôte, Assrs. to Soc. Chem. Ind. in Basle (U.S.P. 1,629,906, 24.5.27. Appl., 29.8.24. Conv., 21.9.23).—See E.P. 238,704; B., 1925, 840.

Manufacture of 2 : 3-aminonaphthoic acid. R. Tobler, Assr. to Soc. Chem. Ind. in Basle (U.S.P. 1,629,894, 24.5.27. Appl., 26.3.26. Conv., 8.4.25).—See E.P. 250,598; B., 1926, 736.

Sulphurised derivatives of naphthols (E.P. 269,971).—See XV.

Photographic desensitiser (G.P. 436,161).—See XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Mildew in cotton goods. IV. Antiseptics and the growth of mould fungi on sizing and finishing materials. L. E. Morris (J. Text. Inst., 1927, 18,

T 99—127. Cf. B., 1925, 166; 1926, 186, 187).—A full statement is made of the biochemical aspects of the problem of the prevention of mildew in cotton goods by means of antiseptics. The efficacy of 32 antiseptics has been examined with a range of common mould fungi, usually 8 species, growing on jellies of flour or starch and agar. The results are combined in one final table, which records the minimum concentration of antiseptic which must be present to ensure that mildew will not appear. Many results are also given as "phenol coefficients"; thus, zinc chloride is only one sixth as effective as phenol, but salicylic acid about $2\frac{1}{2}$ times. The common antiseptics do not appear to be effective in the amounts usually employed. The most efficient new materials tested, for the particular purpose in view, are thallium carbonate and *p*-nitrophenol, but the use of the latter is restricted to acid media on account of the colour of its alkali salts. J. C. Withers.

Chemical analysis of cotton. XIV. Rate of oxycellulose formation. D. A. Clibbens and B. P. Ridge (J. Text. Inst., 1927, **18**, T 135—167).—Measurements have been made of the rate of consumption of oxygen and of change in the properties of the material when bleached cotton is treated with buffered solutions of hypochlorite, of constant active chlorine content but varying acidity or alkalinity. Within the range p_H 5—10, characteristic of technical bleach liquors, the rate of attack depends on the acidity or alkalinity to an extent which accounts for differences encountered in practice which were difficult to explain. It is greatest at the neutral point, but slightly acid solutions are very susceptible to catalytic influences which increase their activity. For example, the traces of chromium left in cotton after treatment with dichromate very greatly enhance the damage by acid hypochlorite. Alkaline hypobromite solutions are more active than equivalent hypochlorites. Dichromate is much more active in the presence of oxalic acid than when an equivalent of sulphuric acid is present. No case has been observed in which slight modification of cotton by one agent rendered it more susceptible to attack by other means. J. C. Withers.

Chemical analysis of cotton. XV. Determination of combined sulphuric and hydrochloric acids in cotton. D. A. Clibbens and A. Geake (J. Text. Inst., 1927, **18**, T 168—174).—When cotton containing traces of sulphuric acid is dried at a high temperature, the acid combines with the cotton and cannot be removed even by boiling with alkali. The cotton is also tendered, and a determination of combined acid therefore affords proof of the cause of damage if suspected to be due to sulphuric acid. A microchemical method for ashing the cotton and converting the acid into barium sulphate is described, and a similar method for hydrochloric acid, which also remains in combination when dried into cotton. The high affinity for basic dyes exhibited by such combinations of cotton and acid is shown to be directly related to the amount of acid fixed. J. C. Withers.

Irish moss mucilage and its determination. P. Haas and B. Russell-Wells (Analyst, 1927, **52**, 265—269).—Irish moss mucilage is a mixture of two substances, one soluble in cold water to form a viscous solution which does not set to a gel on cooling and concentrating, and the other sparingly soluble in cold water dissolving to form a solution which sets to a gel on cooling if the concentration is over 2%. In case a quantitative precipitate results on addition of benzidine hydrochloride solution. The precipitate may be collected, washed with saturated benzidine sulphate solution, and, on warming at 80°, titrated with $0 \cdot 1N$-sodium hydroxide solution with phenolphthalein as indicator. The relative proportion of the two constituents affects the titration but very slightly, and 1 c.c. of $0 \cdot 1N$-sodium hydroxide solution is equivalent to 0·0324 g. of extract. The method can be applied in the presence of other mucilaginous substances, and, with certain modifications, to cod-liver oil emulsion. D. G. Hewer.

Strength testing of chemical pulp. I. Relation between the initial strength of bleached sulphite and the strength properties developed in beating it. G. P. Genberg (Pulp and Paper Mag., 1927, **25**, 537—540).—Detailed results are given of the bursting strength, tear, and freeness of various samples of bleached sulphite pulp before beating and after beating (in a ball mill) for varying periods up to 2 hrs. The results show that the bursting strength and tear in the unbeaten state, hydration qualities, and strength after beating are to a certain extent related; thus, low initial bursting strength and tear indicate an easily hydrating pulp of low strength, whilst high initial strength and tear indicate high strength after beating coupled with easily hydrating properties. Low initial strength and high tear characterise a pulp which hydrates slowly and maintains a high tear during beating; high initial strength and low tear show that the pulp is already somewhat hydrated, and will rapidly develop maximum strength on beating, but will have a low tear. D. J. Norman.

Possibility of obtaining alcohol from cellulose and from wood by way of lævoglucosan. I. Mutti and A. Montalti (Annali Chim. Appl., 1927, **17**, 188—196).—Distillations under the reduced pressure due to a water-pump have been carried out on cotton wool, filter paper, chlorine cellulose, α- and β-celluloses, viscose artificial silk, esparto, and beech- and fir-woods, the carbonaceous residue, tar, aqueous distillate, gas, and loss, and lævoglucosan being determined in each case. The yields of lævoglucosan from wood are so low that the manufacture of alcohol in this way is impracticable. T. H. Pope.

Measurement of the degree of sizing of paper. F. T. Carson (U.S. Bur. Standards, Tech. Paper No. 326, 1926, 20, 703—732).—A brief description is given of 38 sizing tests which have at various times been proposed for measuring the degree of sizing of paper. Most of these tests indicate the resistance of paper to penetration by water and aqueous solutions, *i.e.*, internal sizing, and are not necessarily a measure of the surface sizing of the sheet, *i.e.*, resistance to lateral spread, which is best judged by observing whether or not written characters show any tendency to feather. Penetration tests using ink or interacting solutions are, in general, unreliable owing to selective adsorption of dissolved matter by the cellulose; the best method of this type

appears to be that of Stöckigt using ammonium thiocyanate solution with ferric chloride as a spot indicator. Tests depending upon electrical conductivity are also unsatisfactory, since they measure not only the degree of sizing, but also the resistance of entrapped air, and in many cases the latter has the more pronounced effect. Numerous experiments indicate that the curl method and other methods involving penetration by water, preferably the Bureau of Standards dry-indicator method, are the most reliable for determining the degree of internal sizing of paper. The dry-indicator test is briefly as follows :—Two dye–sugar mixtures of the same colour are made up, one containing about 2% of a water-soluble dye, *e.g.*, Methyl Green or Pontacyl Scarlet, the other containing a pigment insoluble in water; the sugar–water-soluble dye mixture is sprinkled on to the sample through an 80-mesh sieve, and is flanked by the sugar–pigment mixture similarly applied. The sample is then floated on water at a definite temperature, and the time noted until the characteristic colour of the water-soluble dye appears. Comparative tests should be made at the same temperature and relative humidity.

D. J. NORMAN.

PATENTS.

Production of fibrous material for filtering gases and mists. DEUTSCHE GASGLÜHLICHT-AUER-GES.M.B.H., Assees. of H. ENGELHARDT (G.P. 438,554, 8.11.25).—Fibrous material is steeped in a liquid having as nearly as possible the same density as itself, after which it is pressed, and residual liquid is removed by washing with a readily volatile liquid of low density. L. A. COLES.

Treatment of jute fibres. G. PEACE and A. L. CARNEGIE (E.P. 270,150, 18.9.26).—The fibres are treated with a stable emulsion or colloidal dispersion of mineral oil and water, in which the oil is in a very fine state of division, prepared by pulverisation of the materials (*e.g.*, in a colloid mill) in the presence of a small quantity of a stabilising agent, such as 0·1% of sodium oleate. Such emulsions may contain 1—80% of oil, and may be suitably diluted with water. B. P. RIDGE.

Process and apparatus for the industrial treatment of coconuts and their constituents, particularly coconut fibres. Z. VAN DEN BERGH, B. G. H. VAN DER JAGT, and F. A. J. VAN KUYK (E.P. 246,837, 25.1.26. Conv., 2.2.25).—The complete layout of a plant for obtaining spinnable fibre (suitable for sugar bags, fish nets, and the like) and dried copra from fully-ripened coconuts is described. D. J. NORMAN.

Method and apparatus for treating textiles. F. B. VOEGELI (U.S.P. 1,626,438—9, 26.4.27. Appl., [A], 11.6.25, [B], 19.8.25).—(A) Textiles are mercerised by the successive action of hot and cold caustic soda solutions and are then cooled. (B) The cooled fabric passes between a series of stretcher rolls and calender rolls, and having passed through the whole stack, is repeatedly guided back through the same stack to form a number of layers. D. J. NORMAN.

Cleaning textile fibres and fabrics. R. S. HILTNER (E.P. 268,979, 3.5.26).—Wool and other textile fibres are cleaned in one operation, and the by-products obtained in a convenient form, by agitation, preferably in an apparatus working on the counter-current principle, at a temperature not exceeding 71° with an emulsion comprising, for example, equal volumes of water and a liquid which is a good solvent for grease, *e.g.*, petroleum distillates which boil above 75° (preferably between 175° and 225°) or chlorinated hydrocarbons of suitable b.p. Emulsifiers, though usually unnecessary, may be used, also small quantities (0·1—0·5%) of alkalis to maintain neutrality or slight alkalinity; for the latter purpose ammonia, potassium carbonate, or a mixture of potassium and sodium carbonates should be used.

D. J. NORMAN.

Carbonising textile materials. H. LANE and W. MELLOR (E.P. 268,959, 6.4.26).—A carbonisation process, which is rapid and removes both silk and vegetable fibres, involves treating the fabric, previously dried in a current of hot air, with nitric acid vapour. The rags are finally freed from dust and washed.

D. J. NORMAN.

Methods and apparatus for the manufacture of artificial silk. SOC. POUR LA FABR. DE LA SOIE "RHODIASETA" (E.P. 269,377, 22.7.26. Conv., 20.5.26. Addn. to E.P. 238,842; B., 1926, 627).—Apparatus is described for carrying out the process outlined in the original patent. In one arrangement, in which the current of vapour ascends the spinning cell, the cell is jacketed by, *e.g.*, water at 100°, and is in communication both at the top and bottom with an external, vertically disposed condenser. An alternative arrangement, in which the vapour moves downward through the cell, comprises an unheated spinning cell connected at its lower end to a condenser, the circuit being completed by a tube surrounded by a heating device. In both cases the condenser is in communication with a suitable receiver. D. J. NORMAN.

Production of artificial silk. COURTAULDS, LTD., W. H. GLOVER, and C. F. TOPHAM (E.P. 268,455, 7.1.26).—A rapidly-rotating spinning box with reciprocating funnel of the type described in E.P. 23,158 of 1900 is used for collecting and at the same time imparting a twist to artificial silk threads produced by the dry-spinning process. The thread is preferably moistened, optionally with a solution containing a sizing agent, before it reaches the box, and may later be withdrawn therefrom either through the reciprocating funnel with the box rotating (to increase the twist of the thread) or as a cake, in which case its removal is facilitated by heating the box and its contents for about 10 min. at 70°. Means are described for introducing the end of the thread into the rapidly-rotating box by a current of air.

D. J. NORMAN.

Manufacture of flat ribbon-shaped artificial textile fibres from viscose. BRITISH ENKA ARTIFICIAL SILK CO., LTD., Assees. of N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (E.P. 244,446, 24.11.25. Conv., 15.12.24).—Hollow threads of high quality are made by using two coagulating baths, the first of which effects coagulation, leaving an elastic filament of cellulose xanthate, whilst the second decomposes the xanthate and at the same time any gas-producing substances which have been introduced either into the original viscose solution or into the threads coming from the

first coagulating bath, by, for example, passing them through a bath of sodium carbonate solution. The preliminary coagulation may be effected by a weak acid bath, or a bath containing an ammonium salt, spinning with a short spinning trajectory, removing acid from the threads as soon as they leave the bath, or introducing the thread immediately after coagulation into a bath containing an ammonium salt and kept alkaline by additions of, *e.g.*, sodium carbonate. A zinc salt, *e.g.*, 1% of zinc sulphate, may, with advantage, be added to this preliminary coagulating bath. *Example*: Viscose solution containing about 5% of sodium carbonate is spun with a trajectory of about 5 cm. into a bath containing 8% of sulphuric acid and 20% of sodium sulphate, and the filaments are immediately transferred to a bath containing 10% of ammonium chloride and kept faintly alkaline with sodium carbonate. The fibres are finally inflated and fixed in a bath containing about 3% of sulphuric acid. D. J. Norman.

Process for chemically varying artificial silk. Heberlein & Co., A.-G. (E.P. 261,792, 22.11.26. Conv., 21.11.25. Addn. to E.P. 255,453; B., 1927, 103).—The process described in the original patent is applied to artificial silks of cellulosic origin, including cellulose ester or ether silks. D. J. Norman.

Treatment of products containing cellulose acetate. R. Clavel (E.P. 269,605, 10.11.25).—Cellulose acetate yarns that have been delustred either to produce a wool-like effect (cf. E.P. 206,818; B., 1924, 743) or by accident may be relustred by treating them with swelling agents, *e.g.*, formic acid, acetic acid, ethyl alcohol, ethyl acetate, calcium thiocyanate, etc., and then exposing them in a dry condition to the action of substantially dry steam, preferably under slight pressure, under such conditions as preclude the deposition of moisture. Dyestuffs, salts, mordants, etc. may be added to the swelling agents, and, by using suitable resists, lustrous designs on a dull ground may be produced. When a high-boiling solvent has been used in the manufacture of the cellulose acetate filaments, exposure to dry steam is sufficient to restore the lustre. D. J. Norman.

Manufacture of cellulose derivatives. Courtaulds, Ltd., W. H. Glover, and C. Diamond (E.P. 268,552, 10.4.26).—Cellulose derivatives suitable for the manufacture of films and the like are obtained by acetylating an ethylated cellulose which is insoluble in water, dilute alkali, and the common organic solvents, and contains not less than 4% Et nor more than one ethyl group per mol. of $C_6H_{10}O_5$. *Example*: 100 pts. of sulphite wood pulp are impregnated with 21% caustic soda solution, pressed to about 300 pts., and milled for about 2 hrs. at 15—20°. 290 pts. of ethyl sulphate are then added and the milling is continued for 0·5—1 hr. at 15—20°. The mass is then transferred to a suitable vessel and maintained at 15—20° for 3—5 days, preferably in an atmosphere of nitrogen. The resulting ethylcellulose (approximately 15% Et) is washed free from alkali, dried to a moisture content less than 3%, and introduced into an acetylating mixture containing per 100 pts. of ethylcellulose, 2 pts. of sulphuric acid, 200 pts. of acetic anhydride, and 500 pts. of acetic acid. The reaction mixture is stirred continuously, and the temperature initially maintained below 10°; this is subsequently allowed to rise to 15°, at which temperature acetylation is allowed to proceed to completion (10—15 hrs.). The acetate may be precipitated at once or hydrolysed until the desired solubility has been obtained. The final product gives with acetone clear solutions of high viscosity. This process is particularly suitable for preparing cellulose derivatives from wood pulp. D. J. Norman.

Esterification of cellulose. Courtaulds, Ltd., and C. Diamond (E.P. 269,012, 21.6.26).—Cotton cellulose and wood cellulose which have been purified with caustic soda solution are more readily esterified if they are given a preliminary treatment, preferably with agitation, with a relatively small quantity of a phenol, either at the ordinary temperature or at 50—60°. D. J. Norman.

Maturation of alkali cellulose intended for the preparation of viscose artificial silk. "La Soie de Chatillon," Soc. Anon. (E.P. 250,617, 10.4.26. Conv., 10.4.25).—Maturation of alkali cellulose is arrested by removing the oxygen from the atmosphere surrounding it, or accelerated by increasing the amount of oxygen present. In the absence of oxygen the material may be preserved indefinitely without substantial modification of the degree of maturation. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 240,717, 178,152, 14,675 of 1914, and 13,055 of 1914 (cf. B., 1925, 986; 1922, 459 A; 1915, 606).] B. P. Ridge.

Manufacture of wool-like cellulosic material. W. Harrison (E.P. 268,505, 12.2.26).—Cellulosic material, such as cotton, jute, cellulose artificial silk, or the like, is converted into a water-insoluble xanthate as described in, *e.g.*, E.P. 126,174 (B., 1919, 458 A), and is then subjected, preferably after removing by-products with water and optionally after acidifying with acetic acid, to the action of a mild oxidising agent such as air (preferably in the presence of a catalyst, *e.g.*, oxides of iron, cobalt, etc.), ferricyanides, iodine, nitrous acid, or the like, which does not completely remove the carbon disulphide previously combined with the cellulose. The resulting product is not decomposed by dilute mineral acids, heat, or long contact with water; it can be shrunk, and shows a strong affinity for basic colours and certain acid colours, particularly those of the sulphonated basic type. D. J. Norman.

Manufacture of bagasse board. C. W. Mason (U.S.P. 1,627,103, 3.5.27. Appl., 9.9.26).—Bagasse which has been cooked with milk of lime and reduced to a uniform pulp is mixed with 10—20% of raw bagasse and formed into sheets, a number of which are superposed to give a board. The board is finally impregnated with an anti-vermin agent. D. J. Norman.

Stabilisation of plastics. C. E. Burke, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,626,916, 3.5.27. Appl., 24.6.25).—0·5—5% of an ester of phenylglycine is used as a stabiliser for nitrocellulose plastics. D. J. Norman.

Method and apparatus for bleaching pulp. A. D. Merrill (E.P. 269,031, 28.7.26).—A continuous process

is described for bleaching pulp on the counter-current principle, so that the final treatment of the pulp is effected with the strongest bleaching liquor cells. Each cell has associated with it a pulp thickener through which the pulp passes into the succeeding cell by gravity. The consistency of the pulp is approximately the same in each cell, and is maintained above 12%. A bleaching cell is described of such a type that the pulp is given a rotary movement at the bottom of the cell, and is then impelled vertically upwards without rotary movement through a central tube at the lower end of which is a screw conveyor. This process is said to reduce both the time of bleaching and the bleach consumption. D. J. Norman.

Preparing paper pulp. F. K. Fish, jun. (E.P. 255,030, 4.6.26. Conv., 7.7.25).—Pulp of improved fibre and colour is produced with a shorter cooking time by the process described in E.P. 244,788—9 (B., 1926, 871) if sulphur is added either to the chips or to the cooking liquor, so that the reactions between the sulphur and the liquor take place in contact with the chips. D. J. Norman.

Utilising old paper stock. J. E. Plumstead, Assr. to Jessup & Moore Paper Co. (U.S.P. 1,625,279, 19.4.27. Appl., 21.6.26).—A batch of cellulosic material is cooked under heat and pressure and blown into a vessel where it is mixed with a batch of old stock, previously disintegrated with cold water; by this procedure the old stock is cooked and freed from undesirable material. D. J. Norman.

Drying textiles. H. Krantz (U.S.P. 1,629,167, 17.5.27. Appl., 24.3.22. Conv., 15.1.17).—See E.P. 157,425; B., 1922, 459 a.

[Electrolytic] treatment of cellulosic material. K. B. Heberlein (E.P. 270,375, 27.1.26).—See F.P. 610,417; B., 1927, 296.

Heating and drying apparatus [for paper or textiles]. W. B. Fulton (E.P. 270,122, 9.7.26).

[Apparatus for] obtaining fibres from flax, hemp, and similar plants. Fabricord, Inc. (E.P. 269,389, 23.8.26. Conv., 12.6.26).

Winding or reeling artificial silk threads. W. Schulz (E.P. 249,490, 23.2.26. Conv., 17.3.25).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Brightening alizarin pink shades during printing. O. Scheurer (Sealed Note 842, 27.11.95. Bull Soc. Ind. Mulhouse, 1927, **93**, 123). Report by P. Binder (*Ibid.*, 123—124).—Contamination of printing pastes containing alizarin dyes by metallic compounds during printing iron- or chrome-mordanted fabric, and consequent "saddening" of the resulting printed effects, is avoided or corrected by adding 150—200 g. of a gelatinous solution of aluminium hydroxide per 1 kg. of printing paste, before or during printing. Binder suggests that such additions may not always be desirable, and prefers an alternative method previously described (cf. Scheurer; B., 1925, 987). A. J. Hall.

Application of after-chroming dyes to wool and silk. J. Pokorný (Sealed Notes 2453, 21.12.21, and 2470, 15.4.22. Bull. Soc. Ind. Mulhouse, 1927, **93**, 124—125). Report by J. Niederhauser (*Ibid.*, 125—128).—The natural qualities of wool and silk are better preserved if these materials, after dyeing with mordant dyes, are not after-chromed in a boiling solution, but are impregnated with a cold solution consisting (for wool) of 100 c.c. of a 5% solution of a dichromate, 100 c.c. of water, and 10 c.c. of 10% acetic acid, or (for silk) of 100 c.c. of a 2·5% solution of a dichromate and 10 c.c. of 10% acetic acid, and then steamed for 5—15, or 2 min., respectively; the wool is preferably chlorinated before dyeing, and reserves on silk may be obtained by means of fatty substances, dyeing being then effected in a cold solution containing a small quantity of acetic acid. The process yields satisfactory results with Eriochrome Azurol B, Diamond Black PV, Dauphin Blue B, Alizarin Yellow GG and R, and Alizarin Saphirol. Niederhauser reports favourably on the processes, but points out that they are suitable for a selected range of dyes only; the conditions of steaming influence the resulting shades very considerably. A. J. Hall.

Dyeing with Hydron Blue. E. Justin-Mueller (Sealed Notes [a] 2189, 8.7.12; [b] 2191, 25.7.12; and [c] 2207, 15.10.12. Bull. Soc. Ind. Mulhouse, 1927, **93**, 128—131). Report by O. Michel (*Ibid.*, 132—134).—(a) Cotton materials are dyed with Hydron Blue from a liquor containing sodium bisulphite instead of the more usually employed sodium sulphide, the resulting shades having a greater resemblance to indigo, while less sodium hyposulphite is required in the dye liquor. A satisfactory dye liquor consists of 15 pts. of Hydron Blue 40% paste, 35 pts. of caustic soda (d 1·360—1·385), 30 pts. of sodium bisulphite (d 1·360—1·385), and 5 pts. of sodium hyposulphite; it is used at 85°. (b) Brightening of the shade of cotton dyed with Hydron Blue is effected by treatment for 15—20 min. with a cold solution containing 5% (based on the weight of cotton) of sodium bisulphite (d 1·36). It is unnecessary to use a hyposulphite in dye liquors containing Hydron Blue R if the after-treatment described above is used. *E.g.*, cotton is dyed for ¾ hr. at 90° in a solution containing 15% of Hydron Blue R 40% paste and 15% of concentrated sodium sulphide, then treated with sodium bisulphite, allowed to oxidise, rinsed, and then dried. Sulphur dyes may be used together with Hydron Blue R provided that a suitable quantity of sodium carbonate is added to the dye liquor. (c) In dyeing with Hydron Blue, the use of sodium hyposulphite in the dyebath may be replaced by glucose. *E.g.*, a satisfactory dye liquor is prepared with 10% of Hydron Blue R 40% paste, 10% of caustic soda (d 1·385) or 7% of soda ash, and 15% of sodium sulphide crystals, 7% (based on the weight of cotton) of glucose being added in small portions. Dyeing is effected at boiling temperature for 45 min., and the dyed materials are allowed to oxidise during storage for several hours. Alternatively, the dyed material may be directly oxidised and developed by treatment with a cold solution of sodium bisulphite, but the resulting shade has less depth and brightness. Michel reports favourably on the three processes described. The shades obtained with dye liquors containing bisulphite–hyposulphite–caustic soda (a) are deeper and more purple than those obtained under

similar conditions with sodium sulphide–caustic soda–hyposulphite. Superior brightening is obtained (B), but the resulting shades are purer and more purple when sodium perborate is used. When glucose is used in the dyebath (C) copper shades are sometimes obtained.

A. J. HALL.

Treatment [development] of sulphur dyeings with sodium bisulphite. E. JUSTIN-MUELLER (Sealed Note 2265, 21.7.13. Bull. Soc. Ind. Mulhouse, 1927, **93**, 132). Report by O. MICHEL (*Ibid.*, 134).—A process previously described (cf. Justin-Mueller; B., 1921, 145 A) for rapidly developing the full bright tone of fabrics dyed with sulphur dyes is modified, whereby the treatment of the dyed material is effected with a cold solution containing 2—3% of sodium bisulphite (not an alkaline sulphite) of *d* 1·385 for 10—15 min., and washed before drying. A. J. HALL.

Coloured reserves obtained with vat dyes under indanthrene dyes. R. HALLER (Sealed Note 2222, 21.1.13. Bull. Soc. Ind. Mulhouse, 1927, **93**, 134—136). Report by O. MICHEL (*Ibid.*, 136).—Coloured reserves are obtained by printing cotton fabric with a paste containing zinc oxide, manganese salts, anthraquinone, and a vat dye, and then passing it through a dye liquor containing an indanthrene dye; the alkali in the latter liquor penetrates the printed reserve sufficiently to promote development of the vat dye present therein, whilst the indanthrene dye is unable to penetrate simultaneously, and therefore has no soiling effect on the colour of the reserve. *E.g.*, a satisfactory reserve paste for printing on white mercerised cotton fabric consists of 500 g. of a stock solution *A* (consisting of 450 g. of manganous chloride, 500 g. of water, 760 g. of gum, 450 g. of kaolin 50% paste, and 900 g. of zinc chloride), 100 g. of Indanthrene Yellow R 40% paste, 150 g. of zinc oxide 50% paste, and 180 g. of anthraquinone 30% paste. Superior results are obtained when a hyposulphite and potassium sulphite are also added to the coloured reserve, a satisfactory paste consisting of 2000 g. of stock solution *A*, 1000 g. of Hydron Yellow G, 500 g. of anthraquinone, 250 g. of zinc chloride, 280 g. of sodium hyposulphite, and 250 g. of potassium sulphite of *d* 1·385. After printing, the fabric is passed during 30 sec. through a dye liquor prepared with Indanthrene Blue and glucose, a hyposulphite, and an alkali, then washed and soured. Michel reports favourably on the processes.

A. J. HALL.

Fixing basic dyes on vegetable parchment. A. CAILLE (Chim. et Ind., 1927, **17**, 201—202).—The absorption of Methylene Blue by cellulose (filter paper) variously treated with sulphuric acid of parchmentising concentration (*d* 1·68) is dependent on the amount of sulphuric acid chemically combined with the cellulose. Methylene Blue adsorbed by cellulose containing chemically combined sulphuric acid is not easily removed by washing with water, whereas cellulose containing adsorbed sulphuric acid is easily decolorised by washing under similar conditions. A. J. HALL.

Mildew in cotton goods. MORRIS.—See V.

PATENTS.

Increasing the affinity of animal fibres for dyestuffs. CHEMICAL WORKS, FORMERLY SANDOZ (E.P. 245,759, 30.12.25. Conv., 6.1.25).—The material is treated with esterifying agents, *e.g.*, halides of aliphatic, aromatic, or hydro-aromatic carboxylic or sulphonic acids, or the anhydrides of aromatic carboxylic acids, preferably dissolved in organic solvents. *E.g.*, 20 g. of wool are thoroughly worked for 2 hrs. at 40—50° into 250 g. of a 20% solution of *p*-toluenesulphonyl chloride in toluene, squeezed, washed at 60—70° in weak soap solutions, and finally rinsed with cold water.

D. J. NORMAN.

Improving vegetable fibres. HEBERLEIN & Co., A.-G. (E.P. 258,598, 15.9.26. Conv., 16.9.25).—The undesirable effects, viz., unevenness in dyeing and sensitiveness to alkalis, produced when mercerised or unmercerised vegetable fibres are treated with concentrated nitric acid to impart a wool-like character, are removed if the treated fibre is denitrated. Suitable reagents for this purpose are alkali, ammonium, or alkaline-earth sulphides or hydrosulphides, or salts of multivalent metals in their lower stages of oxidation in weak ammoniacal or acid solution, *e.g.*, ferrous chloride, stannous chloride, or the like. D. J. NORMAN.

Dyeing, printing, or stencilling of cellulose acetate materials. BRIT. CELANESE, LTD., G. H. ELLIS, and W. O. GOLDTHORPE (E.P. 269,960, 20.1.26. Addn. to E.P. 242,711; B., 1926, 50).—In the dyeing etc. of cellulose acetate a "secondary solubilising agent" is used in addition to the oily or fatty compounds described in E.P. 219,349 (B., 1924, 906) with or without the "auxiliary solvents" mentioned in E.P. 242,711. The "secondary solubilising agents" are aromatic hydrocarbons of the benzene series, including diphenylmethane etc., naphthalene, chloronaphthalenes, paraffin hydrocarbons, petroleum hydrocarbons, including naphthenes, dodecylene, etc., terpenes, and unsaturated hydrocarbons, *e.g.*, hexene, methyl*cyclo*pentene, 1 : 4-dihydrobenzene, etc.; commercial mixtures such as turpentine, petroleum, solvent naphtha, etc. may also be used. These solvents are claimed to be especially useful when dyeing cellulose acetate with highly insoluble colours, *e.g.*, 4-amino-1-hydroxyanthraquinone (xylene, Turkey-red oil, and olive oil soap), 4-chloro-2-nitro-4′-methoxydiphenylamine (diphenylmethane, sodium sulphoricinoleate, and soap), or 4-nitro-4′-dimethylamino-2-methoxyazobenzene (turpentine, Turkey-red oil, and olive oil soap).

C. HOLLINS.

Dyeing furs, hairs, skins, and feathers. SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (E.P. 270,075, 14.4.26).—The production of blacks on cellulose acetate materials by oxidation of 2 : 4-diaminodiphenylamine (E.P. 258,699; B., 1926, 976) is extended to furs, hairs, skins, and feathers, the shades obtained being grey and brown to black, especially when a copper, chromium, or iron mordant is employed.

C. HOLLINS.

Sulphurised derivatives of phenols and naphthols as mordants (E.P. 269,970).—See IV.

Dyeing cellulose acetate (E.P. 245,758).—See IV.

Treatment of dye effluents (E.P. 262,382).—See XXIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Variables affecting lime used in causticising. J. V. N. DORR and A. W. BULL (Ind. Eng. Chem., 1927, **19**, 558—561).—Various factors influencing the settling rate of calcium carbonate sludge in the causticising of sodium carbonate have been investigated. In one series of experiments the amount of water used for slaking was reduced to one sixth the usual quantity, causing the rate of settlement to increase nearly 12 times. Lime in excess of that normally used has only a slight effect upon the settling rate. Causticity increases and the settling rate decreases with prolonged agitation or when the speed of agitation is increased, and temperatures above 85° are of little benefit. The gradual addition of the soda ash was tried in order to decrease the rate of formation of calcium carbonate, and therefore to increase the size of the particles and the settling rate, but a slow stream of soda ash solution lowered the settling rate, and the causticity was only 92·5%. Hydration of lime with caustic soda instead of water yields an extremely rapid settling carbonate after causticising, but the rate of settlement decreases after 30 min. agitation. Comparison of eleven different limes shows that higher causticity is obtained with a slower settling rate, and that very rapid settling carbonates tend to leave some fine material in suspension, whereas very slow settling gives a bulky precipitate which carries down all the fine material, leaving a clear solution. It is considered that, in causticising, relative particle size persists throughout the reaction, the carbonate being formed at the surface of the hydrated particle and gradually replacing the hydroxide by diffusion in the particle, and, therefore, that coarser hydroxide yields coarser aggregates of calcium carbonate which settle rapidly but leave a turbid supernatant liquor. W. G. CAREY.

***X*-Ray study of limes having different plasticities.** M. FARNSWORTH (Ind. Eng. Chem., 1927, **19**, 583—588).—Marble and precipitated calcium carbonate were burnt in air at various temperatures and marble was calcined in a vacuum furnace. The samples were hydrated with 50% of excess water, and the plasticity of each was tested in a modified form of Emley plasticimeter. *X*-Ray photographs of the oxides and hydroxides were taken with a multiple diffraction apparatus using a Coolidge water-cooled tube with a molybdenum anode. The samples burnt *in vacuo* were more plastic than those burnt in air. The lime samples which give a plastic hydroxide show a face-centred cubic pattern with unit edge 4·79 Å.; the plastic hydroxides give a hexagonal pattern with an axial ratio 1·40. The patterns of the less plastic samples show additional lines corresponding, in calcium oxide films, to strong lines of calcium hydroxide and calcium carbonate films, and, in calcium hydroxide films, to strong lines on the calcium carbonate film. In every case the intensity of these extra lines can be taken as a direct measure of the plasticity of the sample; they are the same for high- and low-temperature burning, but for samples burnt at the higher temperature the intensity is less. The possibility that the calcium hydroxide and calcium carbonate present are the cause of the decrease in plasticity of the hydrated products is suggested. W. G. CAREY.

Effect of particle size on the hydration of lime. F. W. ADAMS (Ind. Eng. Chem., 1927, **19**, 589—591).—Lime was crushed and screened to give nine fractions varying in average diameter between 10 and 0·1255 mm., and were hydrated in an experimental apparatus to produce dry hydrates. After 3 days the samples were tested for moisture, rate of reaction with hydrochloric acid, settling rate, plasticity, and putty volume. It was found that the size of hydrate particles decreases with the size of quicklime particles, and from a finely-ground quicklime a more reactive hydrate with a lower settling rate is obtained. A particle having 10 mm. diameter yields an inferior hydrate with a poor plasticity, whilst one of 5 mm. produces a hydrate with high plasticity. The putty volume generally follows the plasticity figure. W. G. CAREY.

Effect of steam on the decomposition of limestone. E. E. BERGER (Ind. Eng. Chem., 1927, **19**, 594—596).—The rate of calcination of 2 g. samples of graded, washed, and dried limestone in equal currents of air, steam, and helium (115 c.c./min. at 20°) was determined at increasing temperatures from 600° to 1000°, the samples being contained in a platinum cone heated in a quartz tube by a muffle furnace. The rate of calcination was the same for steam and helium, but slightly lower for air, the difference being due, not to chemical action, but to the physical properties of each gas in transferring heat to the sample. Steam may control conditions in the fuel bed by preventing packing of a low-grade coal, by lowering the temperature of the fuel bed, and by lengthening the flame, thus producing a more uniform temperature in the kiln. W. G. CAREY.

Rotary kilns *versus* shaft kilns for lime-burning. R. K. MEADE (Ind. Eng. Chem., 1927, **19**, 597—600).—Limestones which cannot be burnt in a shaft kiln can be dealt with in a rotary kiln provided that the pieces are not greater than 2·5 in. in diameter. In general, rotary kilns are best suited to burn lime: (*a*) where it need not be specially selected; (*b*) where small stone, highly crystalline or very soft limestone, etc. is to be burnt; (*c*) where there are large outputs or operations are continuous; (*d*) where labour is costly and fuel cheap, and where oil or pulverised fuel is used; and (*e*) particularly when power from waste-heat boilers can be utilised. Shaft kilns are preferable: (i) where lime is selected for a particular purpose or when the demand is variable; (ii) where the limestone is hard and compact; (iii) for small operations and when a low first cost is desirable; (iv) when labour is cheap and fuel costly, and when power is not obtainable. The dust loss from shaft kilns is much less than from rotary kilns, so that the former are used where dust is liable to cause a nuisance. W. G. CAREY.

Science and engineering in lime-burning. V. J. AZBE (Ind. Eng. Chem., 1927, **19**, 600—604).—Limestone fed to the kiln should be small and fairly uniform in size. The producer-gas and air must be supplied at a constant rate and under slight pressure, and should be thoroughly mixed before they enter the kiln, otherwise stratification into streams of variable oxygen content will result, with consequent variable kiln temperatures. The kiln must be operated at a definite rate

proportional to the shaft cross-section, and high carbon dioxide, low oxygen, and no carbon monoxide should be found in the waste gases. The drawing should be every 2 hrs., and the same amount of lime should be drawn each time, there being meanwhile no interruption in firing. Kiln temperatures should be controlled by dilution with waste gases which are removed immediately above the decomposition zone where the gas is still hot, and the blower or fan circulating the gas should handle air and recirculation gas mixed, thus lowering the temperature of the gas. W. G. Carey.

Analysis of hydrated lime by a thermo-chemical method. D. F. Richardson (Ind. Eng. Chem., 1927, 19, 625—629).—The difference in the thermal dissociation of calcium and magnesium hydroxides affords a means of distinguishing between them and of ascertaining the degree of hydration in hydrated limes. 1 g. of the sample is heated in an electric furnace out of contact with carbon dioxide and moisture in a tubulated platinum Gooch crucible for 20 min. at successive temperature increments measured by a thermocouple, the charge being removed from the furnace and weighed after each heating. A temperature–loss in weight curve is plotted, and the definite breaks at the decomposition point of each constituent are noted. From these curves the percentage of each constituent is calculated, the magnesium oxide present as hydroxide being obtained by taking the loss in weight at the initial break in the curve, subtracting the free moisture and that combined with sulphur trioxide as gypsum (obtained by analysis), and multiplying the difference by 2·239, which is the magnesium oxide equivalent of the water given off; the second break in the curve is the amount of water combined with calcium oxide, and this multiplied by 3·111 gives the calcium oxide present as hydroxide. The method may also be applied to the carbonate present, but if much magnesium carbonate is present the method becomes inaccurate. Investigations on these lines show that in freshly hydrated limes a small amount only of magnesium oxide is hydrated, but the calcium oxide almost completely. Older hydrates show a much larger percentage of magnesium hydroxide, but with overburnt limes (above 1200°) hydration of magnesium oxide is incomplete, even after 6 years. W. G. Carey.

Preparation of potassium sulphate. K. Kölichen (Mitt. Kali-Forschungsanst., 1919, 13—28; Chem. Zentr., 1927, I., 166).—The greatest yield of potassium sulphate from the interaction of potassium chloride and magnesium sulphate is obtained at 30—40°. The yield is decreased considerably by the presence of sodium chloride owing to the necessity of using more dilute solutions and to the danger of contamination of the potassium sulphate with kieserite; with only 3% of sodium chloride in the solution 20% more water must be used to obtain pure crystals of potassium sulphate. Details of the concentration of the solutions and probable yields are given for the temperature range 0—90° in the presence of varying proportions of sodium chloride. A. R. Powell.

Volume changes on cooling and crystallising magnesium chloride liquors saturated with potassium and sodium chlorides. K. Kölichen (Mitt. Kali-Forschungsanst., 1920, 1—4; Chem. Zentr., 1927, I., 339).—On cooling concentrated solutions of magnesium chloride saturated with potassium and sodium chlorides and removing the crystals the volume of the mother-liquor is 6·5% less than that of the original solution; with less concentrated solutions, the contraction may be as high as 9%. The limiting amount of magnesium chloride in a solution saturated with potassium and sodium chlorides to ensure that no sodium chloride crystallises with the potassium chloride on cooling has been determined; the results confirm the work of Boeke (Kali, 1909, [13, 14]), but vary widely from those of Feit and Przibylla (*ibid.*, 1909, [18]). A. R. Powell.

Evaporation of mother-liquors [of the potash industry] at a definite temperature and a definite reduced pressure. Contraction phenomena on diluting lyes and salt solutions. W. Holle (Mitt. Kali-Forschungsanst., 1920, 7—8, 9—10; Chem. Zentr., 1927, I., 338).—The mother-liquors from the crystallisation of potassium chloride may be evaporated at 70° and 625 mm. without danger of precipitation of artificial carnallite, but the evaporation cannot be carried as far as is possible when working under pressure. Dilution of solutions of salts or caustic alkalis with a definite volume of water is accompanied by a contraction in volume and an evolution of heat; conversely, therefore, evaporation of the solution is accompanied by an expansion in volume and absorption of heat. A. R. Powell.

Continuous cooler for potassium chloride liquors. Ritter (Mitt. Kali-Forschungsanst., 1920, 11—20; Chem. Zentr., 1927, I., 164—165).—Rapid and continuous cooling of hot potassium chloride liquors leads to the production of small crystals which can be washed satisfactorily only in vessels provided with stirring gear or elevating screws. Cooling apparatus using cold water as the cooling medium effects more rapid cooling than that employing a counter-current of air, and coolers containing a series of baffles over which the hot liquid trickles in thin stream are more difficult to operate efficiently than those of the usual tower type. A. R. Powell.

Cross-current air-stream cooler for potash liquors. Jung (Mitt. Kali-Forschungsanst., 1920, 41—44; Chem. Zentr., 1927, I., 165).—In this type of cooler the liquid falls in thin streams down a long tower without baffles, whilst a current of cold air is blown through laterally. A 25% better vacuum tin the condenser is produced, thus saving up to 30% of the fuel costs. Operation is continuous, the crystals being withdrawn automatically from the bottom of the cooler without interruption of the flow of liquid. A. R. Powell.

Tower coolers and paddle coolers for potash liquors. Schade and Winter (Mitt. Kali-Forschungsanst., 1920, 45—54; Chem. Zentr., 1927, I., 165—166).—Tower coolers give efficient results with potassium chloride liquids, but have the disadvantage that they can be worked only intermittently owing to the necessity of removing the crystals from time to time. Paddle coolers do not suffer from this disadvantage, as they can be run continuously while the crystals are raked out from the bottom of the liquid. They operate on the

principle of a paddle wheel rotating half submerged in the hot solution, while a counter-current of cold air is driven across the surface. As the wet paddles rise from the liquid the air stream causes rapid evaporation of some of the moisture on their surface with consequent cooling of the whole volume of liquid. A. R. Powell.

Examination of Przibylla's tartaric acid method for the determination of potassium. E. Borsche (Mitt. Kali-Forschungsanst., 1920, 61—83; Chem. Zentr., 1927, I., 149—150).—The tables given by Przibylla for calculating the results from the titration figures are applicable only to materials containing 80—100% KCl. For the determination of potassium in mixed fertilisers and by-products of the potash industry special tables must be compiled from comparison of the results obtained by a standard gravimetric method with those obtained from the tartrate titration on each type of material. A. R. Powell.

Determination of the specific heat of various solutions obtained in the potash industry. Küpper (Mitt. Kali-Forschungsanst., 1920, 85—90; Chem. Zentr., 1927, I., 150).—For solutions containing relatively small amounts of magnesium chloride the sp. heat may be calculated by dividing the weight of water present in 1 litre of the solution by its sp. gr. The results are correct to $\pm 0\cdot 05$. The errors increase with a rise in the magnesium salt content of the solution, but if this is known, corrections can be applied by the use of a table given. Magnesium compounds have a much greater effect than compounds of the alkali metals in reducing the heat of the solutions obtained in working up the Stassfurt potash deposits. A. R. Powell.

Specific heat of carnallite and the heat of dissolution of carnallite and potassium chloride in leach liquors. A. Küpper (Mitt. Kali-Forschungsanst., 1920, 99—111; Chem. Zentr., 1927, I., 338).—The sp. heat of carnallite is 0·371, and of carnallite containing 16% of potassium chloride 0·312. The heats of dissolution of potassium chloride and of carnallite in the leach liquors at 105—110° are very small. From these data it follows that the quantity of leach liquor and the heat required for leaching are lower the lower the magnesium chloride content of the leach liquor. Too weak a leach liquor, however, results in a final mother-liquor rich in potassium chloride, which requires more heat for its evaporation and which deposits large quantities of artificial carnallite. A. R. Powell.

Preparation of potassium sulphate by Hargreaves' process. K. Hepke (Mitt. Kali-Forschungsanst., 1920, 91—97; Chem. Zentr., 1927, I., 166—167).—Potassium sulphate of 98% purity is obtained by heating potassium chloride containing a maximum of 0·5% of sodium chloride at a dull red heat for 12 days in a current of air containing sulphur dioxide. The product is a fine white power entirely free from acid. By passing the issuing gases through water a solution of hydrochloric acid of *d* 1·05—1·12 is obtained practically free from sulphur dioxide. A. R. Powell.

The potassium-magnesium sulphate process. K. Kölichen and C. Przibylla (Mitt. Kali-Forschungsanst., 1920, 113—131; Chem. Zentr., 1927, I., 338—339).—For the production of potassium magnesium sulphate (schönite) from by-products of the potash industry the magnesium sulphate solution used should be as concentrated as possible, and relatively free from sodium chloride so as to avoid contamination of the crystals of schönite with double sulphates of magnesium and sodium (astrakhanite and glaserite). Working with concentrated solutions at 25°, the product obtained contains 20—30% more of the potassium and 8—10% more of the sulphate ion in the reacting solutions than is obtained by the usual process, and the mother-liquor contains about 200 g./litre of magnesium chloride, instead of the usual 130 g./litre, and is therefore more suitable for direct use in leaching further quantities of carnallite. A. R. Powell.

Removal of slimes from hot, crude [carnallite] liquors by filtration in the Kelly press. Ratig (Mitt. Kali-Forschungsanst., 1921, 1—12; Chem. Zentr., 1927, I., 503).—The Kelly press in the same form as is used in the sugar industry may be employed for filtering the hot leach liquors from the extraction of carnallite. The slimes so obtained have a much lower potassium content than those obtained by the use of the old suction filter, but the sodium chloride content is also lower. The construction and method of working the press are described and its advantages discussed.

A. R. Powell.

Filtration of crude [potassium chloride] solutions in the Sweetland filter-press. Rinck (Mitt. Kali-Forschungsanst., 1921, 13—16; Chem. Zentr., 1927, I., 504).—Leach liquors from the extraction of carnallite may, after the addition of a small quantity of water, be filtered through a Sweetland press in which fine brass or phosphor-bronze gauze replaces the usual filter-cloths. The life of the metal cloths is about 3—4 months. The effluent from the press contains slightly more sodium chloride and magnesium sulphate than that obtained from settling tanks, but the filtration process is the cheaper to operate.

A. R. Powell.

Rate of dissolution and displacement of sylvine and rock salt from natural sylvinite and "hard salt." H. Keitel (Mitt. Kali-Forschungsanst., 1922, 95—127; Chem. Zentr., 1927, I., 504—505).—Potassium chloride dissolves more rapidly than sodium chloride from mixtures of rock salt and sylvine at all temperatures, and the rate of dissolution of potassium chloride in saturated brine is independent of the temperature; rock salt, however, dissolves very slowly in saturated potassium chloride solution at the ordinary temperature, but much more rapidly at higher temperatures and in solutions containing magnesium chloride or sulphate. Sodium chloride has a retarding influence on the crystallisation of potassium chloride, probably owing to the formation of a more soluble double salt. The results indicate that extraction of potassium salts from "hard salt" (a mixture of sylvinite, kieserite, and rock salt) on the counter-current principle is not satisfactory owing to the danger of formation of a finely-divided crystalline slime of sodium chloride; leaching on the direct-current principle, on the other hand, produces a hot solution with the maximum content of potassium chloride, and,

therefore, results in a greater output from the same plant. A. R. Powell.

Cold-leaching of carnallite. A. Küpper (Mitt. Kali-Forschungsanst., 1922, 30—41; Chem. Zentr., 1927, I, 503—504).—Cold-leaching of carnallite is a two-stage process. In the first stage the crude salt mixture is leached with the liquor from the second stage to remove the greater part of the magnesium chloride and leave a residue rich in potassium chloride; in the second stage this residue is leached with a fresh quantity of leach liquor. In both stages the leaching is conducted on the direct-current principle, using solutions unsaturated with respect to sodium chloride. With this procedure the most rational method for the recovery of the potassium appears to be that involving crystallisation of the sulphate after addition of the necessary quantity of mother-liquor from the sulphate plant. A. R. Powell.

Leaching [crude carnallite] with final mother-liquors [from previous operations]. H. Keitel and Gerlach (Mitt. Kali-Forschungsanst., 1922, 189—196; Chem. Zentr., 1927, 1, 504).—For leaching crude carnallite with concentrated magnesium chloride liquors the mineral must be finely powdered and leached on the counter-current principle, the settling tank must be well heat-insulated, and the solution must be separated from the residual potassium chloride at the highest possible temperature to prevent, as far as possible, any reversion of this salt to carnallite. On cooling the clear solution, artificial carnallite is deposited and the residual mother-liquor is considerably enriched with lithium and bromine salts. The process is not applicable to material containing tachydrite, as enrichment of the mother-liquor with calcium salts interferes in the subsequent operations. A. R. Powell.

Leaching [carnallite] to obtain the usual mother-liquor. A. Küpper and Althammer (Mitt. Kali-Forschungsanst., 1924, 1—15; Chem. Zentr., 1927, I, 505).—The process described is a compromise between the usual leaching process and that of Keitel and Gerlach (cf. preceding abstract). The leach liquor has a composition approximating to that of a carnallite solution, and extracts about half the potassium chloride from the normal charge of carnallite, together with all the magnesium chloride, so that after crystallisation the final mother-liquor is similar to that obtained in the usual process. This procedure, with suitable coupling of the sulphate and potassium chloride operations, halves the steam consumption, reduces considerably the washing process, obviates the necessity for using the double sulphate method of working up end liquors, avoids slime troubles, and gives a good yield of kieserite. A. R. Powell.

Manufacture of Glauber's salt. K. Kölichen and W. Althammer (Mitt. Kali-Forschungsanst., 1923, 71—86; Chem. Zentr., 1927, I, 505—506).—A high yield of Glauber's salt with a minimum number of operations is obtained by treating magnesium sulphate mother liquors with a dilute brine (200 g./litre of sodium chloride) or with a brine saturated at 80° with astrakhanite (cf. following abstract). Anhydrous sodium sulphate is prepared from Glauber's salt by one of the following three methods: (*a*) heating at 60°, (*b*) heating at 60—70° with sodium chloride, and (*c*) heating at 70° with astrakhanite. The yields obtained by these methods are 42%, 90%, and over 99% respectively. A. R. Powell.

Preparation of astrakhanite. K. Kölichen and W. Althammer (Mitt. Kali-Forschungsanst., 1923, 55—69; Chem. Zentr., 1927, I, 505).—Astrakhanite ($MgSO_4,Na_2SO_4,4H_2O$) may be prepared by treating a solution of magnesium sulphate with brine, by treating the mother-liquor from the manufacture of Glauber's salts with mother-liquors from the potash industry containing magnesium chloride, or by hydrating kieserite ($MgSO_4,H_2O$) in the presence of sodium sulphate or of the mother-liquors from the crystallisation of Epsom salts. The yields by all the methods are approximately the same, but the fuel and labour costs are lowest in the last-named process. A. R. Powell.

Iodine content of the salt from Rumanian rock-salt mines. D. Radulescu and V. Georgescu (Ann. Mines Roumania, 1925, No. 14, Publ. Inst. Chim. Univ. Cluj, 1926, 2, 156—160; Chem. Zentr., 1927, I, 339).—The iodine content of the salt from Rumanian mines varies with the nature of the deposit, large colourless crystals of rock salt containing only minute traces of iodine and black, earthy, crystalline masses being relatively rich in this element. The richest material is that containing a high content of gypsum, but the iodine content of this is only a little more than 1 mg./kg., and is, therefore, of the same order of magnitude as that of the salts recovered by the evaporation of sea-water. A. R. Powell.

Apparatus for the manufacture of calcium hydrogen sulphite solutions. O. Giller (Papierind. [Russia], 1923, 2, 625—633; Chem. Zentr., 1927, I, 167).—Solutions of calcium hydrogen sulphite for use in the cellulose industry may be efficiently prepared by treating milk of lime with gases containing sulphur dioxide in a tower divided by horizontal and vertical wooden partitions into a series of chambers through which the gases pass consecutively. Every division of the tower is accessible for cleaning purposes without disturbing the remainder, and the dividing partitions between the chambers are so arranged that the gases keep the liquid agitated, thus avoiding the necessity of using mechanical stirrers. A. R. Powell.

Relation of bleach concentration to specific gravity. M. N. Roberts (Pulp and Paper Mag., 1927, 25, 610—611).—For bleach liquors made from bleaching powder and containing up to 28·34 g./litre of available chlorine (*d* of liquor at this concentration 1·1337), the available chlorine content varies directly as the sp. gr. of the liquor. Milkiness, unless excessive, has but little effect on either of these factors. D. J. Norman.

Solubility of copper sulphate, malachite, and Burgundy precipitate in ammonia, ammonium carbonate and bicarbonate. E. B. Holland and G. M. Gilligan (J. Physical Chem., 1927, 31, 728—741).—The solubility of malachite, washed Burgundy precipitate, and copper sulphate in ammonia solution increases in the order given, and the solubility of the copper in

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ammonia is in the same order. The cuprammoniums soluble in ammonia solution are unstable on dilution and also in relatively high concentrations of ammonia. The amount of solvent required to counteract the effects of dilution is in the reverse order of the solubilities. With ammonium carbonate as solvent, the order of increasing solubility is Burgundy precipitate, malachite, and copper sulphate, but is more uniform than in the former case, whilst the solubility of the copper in terms of ammonia is practically constant. The corresponding cuprammoniums are more stable on dilution, copper sulphate only requiring a small excess of solvent to prevent hydrolysis. With ammonium bicarbonate the order of solubility is Burgundy precipitate, malachite, and copper sulphate. The bicarbonate, however, is less effective as a solvent than the carbonate. In the case of copper sulphate more bicarbonate than carbonate is required to prevent hydrolysis. L. S. THEOBALD.

Gravimetric determination of chromium with mercury ammonium chloride ($HgClNH_2$). W. PUMM (Collegium, 1927, 202—205. Cf. Solaja; B., 1925, 480).—200—300 c.c. of the chromium solution containing 0·1—0·15 g. Cr_2O_3 are treated with 5 g. of ammonium nitrate and sufficient 20% ammonia solution to produce a distinct turbidity. A solution of 2 g. of mercurous chloride and 3 g. of ammonium chloride in 30 c.c. of water is added, the mixture is stirred, and to it on the formation of a precipitate 2—3 c.c. of 20% ammonia solution are added drop by drop. After being well stirred, 50 c.c. of 96% alcohol are added, and the mixture is kept for 14—16 hrs. It is then twice decanted with 100 c.c. of distilled water, filtered, and washed with 80 c.c. of cold 2% ammonium nitrate solution to remove chlorine. The precipitate is transferred to a filter with 100 c.c. of alcohol (1 : 5), dried, ignited, cooled, and weighed. The errors in a series of tests were less than 0·3% compared with 1·1 % by the ordinary gravimetric method. D. WOODROFFE.

Determination of sulphur in pyrites by the Lunge-Berl method. KASTNER (Ann. Chim. Anal., 1927, [ii], 9, 134—136).—The difficulty of freeing the ferric oxide from the last traces of sulphur in this determination is overcome as follows: 50 c.c. of solution representing 0·5889 g. of pyrites are diluted to 100—125 c.c., neutralised by ammonia solution (15—16 c.c.), and, after settling, the clear liquid is filtered and the precipitate washed with hot water, the 150—170 c.c. of filtrate being removed. The hydrated ferric oxide is dissolved on the filter in boiling hydrochloric acid, and the filter washed 3 or 4 times with hot water. The excess of hydrochloric acid is neutralised, and ferric hydrate precipitated by the same volume of ammonia solution as before. This liquid is filtered into the first filtrate. Practically all occluded sulphur is thus removed. D. G. HEWER.

Silica gel. A. J. C. DE WAAL (Chem. Weekblad, 1927, 24, 274—284).—The preparation, properties, and applications of silica gel are described, with full references to the patent and technical literature. S. I. LEVY.

Determination of hydrogen sulphide in gas. CIOCHINĂ.—See II.

Action of salt solutions on alloys. SAMUEL.—See X.

Lime and limestone in the basic open-hearth process. HERTY.—See X.

Potentiometric titrations. HAHN and others.—See XI.

Importance of potash salts for soils. NOLTE.—See XVI.

Lime in the beet sugar industry. SHAFOR.—See XVII.

Use of lime in butter making. OVERMAN.—See XIX.

Use of lime in water softening. HOOVER.—See XXIII.

Lime-treatment of cannery wastes. WARWICK.—See XXIII.

PATENTS.

Production of sulphuric acid in a reaction tube. F. BRANDENBURG (G.P. 437,727, 20.5.25).—A mixture of sulphur dioxide, air, and steam, with or without nitric acid vapour, is passed through a spiral reaction tube or through a straight tube provided with spiral baffles so as to obtain an intimate admixture of the gases. The process may be utilised for the direct production of sulphates; in this case a finely-divided salt of the metal is introduced into the gas stream so as to react with the sulphuric acid in the nascent state. A. R. POWELL.

Production of sulphuric acid. METALLBANK U. METALLURGISCHE GES. A.-G., Assees. of T. SCHMIEDEL (G.P. 437,782, 17.10.24. Addn. to G.P. 421,786; B., 1926, 319).—Steam or atomised water or dilute acid is added to a mixture of the reaction gases with oxides of nitrogen, under conditions such that formation of sulphuric acid of sufficiently high concentration to dissolve the oxides of nitrogen is delayed as long as possible, but strong acid is eventually formed. L. A. COLES.

Production of hydrochloric acid and magnesia from magnesium chloride. CHEM. FABR. WOLKRAMSHAUSEN G.M.B.H., and M. HELBIG (G.P. 438,177, 5.1.24).—Hydrated magnesium chloride, especially that containing 2—3 mols. of water per mol. of magnesium chloride, is heated suddenly at 360—400°, and a mixture of the magnesium oxychloride formed thereby with magnesium hydroxide is heated at about 500°, or, alternatively, a mixture of magnesium chloride and magnesium hydroxide is heated rapidly at about 500°, *e.g.*, in a rotating furnace or in a muffle furnace fitted with a mechanical stirrer. L. A. COLES.

Manufacture of hydrochloric acid and magnesia [from magnesium chloride]. VER. FÜR CHEM. U. MET. PROD. (G.P. 436,241, 11.3.25).—Magnesium chloride or oxychloride is heated in a current of steam in a rotating tube furnace provided with adjustable external and internal sources of heat. A. R. POWELL.

Preparation of alkali sulphate and hydrochloric acid from alkali chloride. C. UEBEL (G.P. 437,371, 12.5.25).—A molten mixture of the alkali chloride and sulphate is treated with oxygen, steam, and sulphur dioxide, further quantities of alkali chloride being fed in from time to time from a feed chamber in which it is preheated by the gases produced in the reaction. A. R. POWELL.

Production of phosphoric acid and hydrogen. I. G. Farbenind. A.-G. (G.P. 438,178, 21.11.24).—Phosphorus is treated at a high temperature with a limited supply of steam, with or without the addition of air or oxygen, so that lower phosphorus oxides or acids are formed in addition to hydrogen and phosphoric acid, and oxidation to phosphoric acid is completed at a lower temperature, using air or other oxidising agent for the purpose. L. A. Coles.

Colloidal solutions of silicic acid. M. Prätorius and K. Wolf (F.P. 612,486, 8.3.26. Conv., 18.3.25).—Sodium silicate solutions are electrolysed in a cell in which the anode is separated from the cathode by at least three porous diaphragms made of clay, asbestos, wool, or the like. A. R. Powell.

Manufacture of active silica. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 270,040, 26.2.26).—A decomposable silicon compound is added to an acid at a low temperature in such proportion that a sol with a non-alkaline reaction is produced, containing at least 9 g. of silica/100 c.c. of liquid. W. G. Carey.

Synthetic production of ammonia. G. F. Uhde (E.P. 253,122, 2.6.26. Conv., 4.6.25).—The catalyst is made by heating a solution of potassium ferrocyanide with a hot dilute solution of aluminium chloride, which after evaporation yields a water-insoluble complex compound containing aluminium, and which does not retain water. W. G. Carey.

[Apparatus for] ammonia synthesis. Soc. Chim. de la Grande Paroisse, Azote & Prod. Chim. (F.P. 611,417, 2.6.25).—In an apparatus for the synthesis of ammonia consisting of several contact chambers working in parallel, an even distribution of the gas mixture is obtained by arranging for a considerable fall in pressure either before or after the gas passes over the contact mass or by using a pressure pump having as many cylinders as there are contact chambers. A. R. Powell.

Separate recovery of monopotassium phosphate and potassium nitrate from their mixed solutions. I. G. Farbenind. A.-G., Assees. of R. Griessbach and K. Röhre (G.P. 438,229, 1.10.25).—A hot solution saturated or nearly so with both salts is diluted and cooled, and, after removing crystallised potassium nitrate, the mother liquor is concentrated until again nearly saturated with potassium nitrate. Monopotassium phosphate which crystallises from the hot solution is removed, and the process is repeated, if necessary, with the residual liquor. L. A. Coles.

Manufacture of monoammonium phosphate. E. L. Larison, F. F. Frick, and R. J. Caro, Assrs. to Anaconda Copper Mining Co. (U.S.P. 1,628,792, 17.5.27. Appl., 3.5.26).—A gas containing ammonia is scrubbed with a solution of phosphoric acid until the ratio of NH_3 to P_2O_5 is 1 : 2·8 by wt. W. G. Carey.

Removal of phosphorus from metal-bearing solutions. R. H. Stevens, G. C. Norris, and W. N. Watson (E.P. 269,778, 23.8.26).—An acid solution containing at least 1 pt. by wt. of ferric iron to 1 pt. of phosphoric pentoxide is neutralised and heated, and the precipitate of ferric phosphate removed. If vanadium is to be recovered a soluble ferrous compound is added to reduce the vanadium to the quadrivalent condition. W. G. Carey.

Manufacture of iron carbonyl. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 269,625, 11.1.26).—Iron is treated in a reaction vessel with a very slow stream of carbon monoxide at an elevated temperature and pressure, and the reaction gases are brought into contact with cooler surfaces so near to the reaction zone that the gas containing iron carbonyl is conveyed thereto by diffusion or convection, the iron carbonyl separating on these surfaces at the pressure prevailing in the reaction zone. W. G. Carey.

Purification of industrial zinc sulphate solutions preparatory to electrolysis. R. H. Stevens, G. C. Norris, and W. N. Watson (E.P. 269,776, 23.8.26).—Nickel is removed from zinc sulphate solutions by the addition of zinc dust to the neutral or slightly acid heated solution in which soluble copper and arsenic compounds are present. The precipitation and separation of the nickel are effected in the absence of certain specified organic substances which inhibit the zinc–copper–arsenic precipitation, and the reaction is performed in vessels, preferably lead-lined, which do not expose organic substances to the solution. W. G. Carey.

Manufacture of copper sulphate. L. and M. Meyer (Huttenwerke Tempelhof A. Meyer) (E.P. 264,510, 12.1.27. Conv., 13.1.26).—Alloys of copper with tin, lead, or antimony, or metallurgical ashes and scraps containing copper, are converted into sulphides, the mixture is roasted, and the copper oxides and sulphate are separated by leaching with sulphuric acid. W. G. Carey.

Production of lead oxide or carbonate from lead sulphate. R. Daloze (F.P. 612,345, 1.3.26).—Lead sulphate or neutralised lead chamber mud is digested with a saturated solution of sodium or ammonium chloride, and the clarified liquid is treated with ammonia, ammonium carbonate, or sodium carbonate. The filtrate from the lead hydroxide or lead carbonate is distilled with lime for the recovery of the ammonia, which is used again in the process. A. R. Powell.

Purification of fluorspar. Comp. de Prod. Chim. et Electrométallurgiques Alais, Froges, et Carmague (F.P. 612,142, 26.6.25).—Finely-powdered fluorspar is heated with alkali hydroxide solution under pressure to remove silica. A. R. Powell.

Manufacture of alkali sulphates. Rhenania Verein Chem. Fabr. A.-G., Zweigniederlassung Mannheim, Assees. of F. Rüsberg (G.P. 433,351, 30.3.22. Addn. to G.P. 405,922; B., 1925, 169).—Solutions of ammonium sulphate are treated with an excess of aqueous sodium chloride solution in the presence of ammonia, and sodium sulphate separates from solution. The mother-liquor containing sodium chloride is utilised in the ammonia–soda process. S. Pexton.

Central feed for mechanical sulphate kiln. Rhenania Verein Chem. Fabr. A.-G., Assees. of F. Böhm (G.P. 433,352, 26.4.25).—A rotating kiln furnace is provided with salt hoppers closed from underneath

by a plate which revolves with the kiln. Combined with the plate is a screw conveyer which forces the salt into the furnace. S. PEXTON.

Converting alkali sulphides into alkali sulphites. WEST VIRGINIA PULP & PAPER CO., Assees. of V. DREWSEN (E.P. 260,552, 29.7.26. Conv., 29.10.25).—A hot concentrated aqueous solution of alkali sulphide is agitated and mixed with air in a closed mixing machine in the presence of solid comminuted material to give surface, such as solid anhydrous sodium sulphite, at 120—160°. The sulphide may be recovered from spent sulphite liquor by concentration. W. G. CAREY.

Preparation of hyposulphites and related compounds. I. G. FARBENIND. A.-G., Assees. of H. KAUTZKY and H. THIELE (G.P. 436,997, 16.1.24).—Solutions of sulphur dioxide, acid sulphites, or their ketone or aldehyde compounds are treated with silicon compounds derived from a lower oxidation stage than corresponds with silica. A. R. POWELL.

Decomposition of compounds [*e.g.*, alkali silicates]. H. MEHNER (Canad. P. 262,339, 11.8.25).—Sodium or potassium silicate is heated with carbonaceous materials, and the mixture of carbon monoxide and sodium or potassium vapour so obtained is burnt to produce the corresponding alkali carbonate. A. R. POWELL.

Preparation of metal hydroxides for use as catalysts. I. G. FARBENIND. A.-G., Assees. of R. GRIESSBACH, W. MICHAEL, and K. RÖHRE (G.P. 436,671, 16.9.24).—Hydroxides of metals for use as catalysts are prepared by precipitation of solutions of a salt of the metal with an alkali, and are washed with a solution having the same p_H value as the mother-liquor after precipitation. A similar procedure may be applied to the production of hydroxides of an acidic nature. A. R. POWELL.

Calcining magnesite. MAGNESIT-IND. A.-G. (Austr. P. 104,404, 7.3.24).—Magnesite is calcined in a rotary kiln, the temperature of which is so controlled that the material in the region nearest to the source of heat is dead-burnt. In the more remote parts of the kiln there are then zones in which the magnesite is calcined to such a stage that the product has the properties requisite for the use to which it is to be put. Means are provided for removing the magnesia from these zones without admixture of the dead-burnt product. A. R. POWELL.

Production of granulated dust-free nitrate of lime. NORSK HYDRO-ELEKTRISK KVAELSTOFAKT. (E.P. 264,480, 4.1.27. Conv., 14.1.26).—Crushed calcium nitrate is sifted, and the dust is moistened and subjected to continuous motion at ordinary temperature, or without moistening at about 80°. W. G. CAREY.

Separating hafnium and zirconium. N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 258,243, 26.8.26. Conv., 12.9.25. Addn. to E.P. 235,217; B., 1925, 713).—A solution of zirconium and hafnium phosphates in concentrated sulphuric acid is treated with one or more salts of hafnium and zirconium in concentrated sulphuric acid, with the addition of water as a precipitant. The phosphate precipitated contains relatively more hafnium than the original concentrated acid solution, and is re-treated. The oxides required for addition may be prepared by treating the mixture of hafnium and zirconium phosphates with hydrofluoric acid, and then precipitating with a base. C. A. KING.

Gas burners and heating apparatus particularly for hydrogen and nitrogen generators. L'OXYHYDRIQUE FRANÇAISE (G.P. 433,519, 3.3.25. Conv. 10.2.25).—A plant for producing hydrogen by the reduction of steam with iron is built within a heat-recuperating chamber provided with a burner at the top. During the reduction stage gases from the reaction chamber are burnt and the heat is stored round the reaction chamber. In the flue leading from the combustion chamber is built a steam superheater which communicates with the reaction chamber. S. PEXTON.

Liquid air oxygen-producing plant. O. SIMONIS, and LIQUID AIR, LTD. (E.P. 269,661, 21.1.26).—A constant pressure of 5—10 atm. is maintained, irrespective of variations in consumption, in the rectification column of a liquid-air plant by an automatic control on the delivery, and the oxygen is discharged under pressure from the column to a pressure reservoir fitted with a safety valve and a regulator for ensuring constant gas delivery irrespective of varying pressure in the reservoir. Regulator valves are fitted to the oxygen and nitrogen outlets to predetermine the working pressure of the apparatus. W. G. CAREY.

Recovery of sulphur from minerals. H. J. VEILLET (F.P. 612,732, 13.3.26).—The powdered mineral is heated in a furnace containing a number of circular, superimposed hearths provided with rabbles so arranged that the substance passes from the centre to the periphery and from the periphery to the centre on alternate hearths. The furnace is heated externally and the charge is protected from oxidation by the air. A. R. POWELL.

Production of coloured sulphur castings. P. SCHOLZ (G.P. 436,170, 14.3.24).—Sulphur, with any suitable addition substance other than the colouring matter, is heated at 200—300° or higher, the mixture well stirred and allowed to cool to the casting temperature (140—180°), the colouring matter stirred in, and the product cast as usual. A. R. POWELL.

Sulphur burner. H. F. MERRIAM, Assr. to GENERAL CHEMICAL CO. (U.S.P. 1,629,352, 17.5.27. Appl., 3.12.23).—The burner chamber has an inlet for feeding sulphur, and another inlet for compressed air, also a device for preventing the escape of gases or air from the sulphur feed-inlet. W. G. CAREY.

Automatic feed for sulphur burners or distillation retorts. RAFFINERIES INTERNAT. DE SOUFRE (F.P. 607,632, 17.3.25).—Solid sulphur is fed continuously on to trays over which hot gases and vapours from the burner pass. The sulphur melts and flows in a regular manner into the furnace or distillation apparatus. S. PEXTON.

Producing smoke for forming smoke screens and like purposes. R. THRELFALL, A. A. KING, and J. G. CLARKE (E.P. 269,979, 27.1.26).—A smoke shell or bomb contains a charge of white phosphorus and red

phosphorus emulsified with gelatin, which latter protects the phosphorus from rapid combustion, and so prevents dissipation of the cloud due to excessive heating of the surrounding air. W. G. Carey.

Production of sodium aluminate. Aluminum Co. of America, Assees. of J. B. Barnitt (E.P. 264,823, 11.1.27. Conv., 21.1.26).—See U.S.P. 1,616,674; B., 1927, 388.

Separation of soluble substances (F.P. 612,993).—See I.

Compressing viscous materials (F.P. 606,190).—See I.

Electrolytic separation of fused salts (E.P. 269,749).—See XI.

VIII.—GLASS; CERAMICS.

Behaviour of fluoride ingredients of glasses and enamels. I. G. Agde and H. F. Krause (Z. angew. Chem., 1927, **40**, 525—533).—A series of enamels containing either sodium, calcium, or aluminium fluoride as opaliser, was melted rapidly, chilled, and reheated to produce opacity. In some cases the glass became opalescent on first cooling, in others the temperatures of reheating required ranged from 510° to 770°. In all cases the opalising medium was a precipitated solid, either in the form of small globules, crystal skeletons, or cubes. The precipitate obtained in the sodium and aluminium fluoride series had a refractive index of 1·334—1·360, and was soluble in water and acids, indicating sodium fluoride (n 1·334). In the calcium fluoride series, the value of n (1·40—1·44), insolubility in water, and slow solubility in acids proved the opalising medium to be calcium fluoride (n 1·435). All deposits were optically isotropic. With little supercooling there was a formation of small globules, but greater cooling gave either crystal aggregates or cubes depending on the concentration of the fluoride. The presence of alumina in the glass tended to reduce the size of the particles. Best results were obtained commercially with glasses which were clear on first chilling and which were subsequently so reheated as to give fine crystals. Large crystals, besides giving a weak and uneven opalescence, adversely affected the physical properties of the glass, such as elasticity. A. Cousen.

Silica glass. H. George (Compt. rend., 1927, **184**, 1046—1047).—In the production of opaque silica glass by fusion of pure siliceous sand in the electric furnace with carbon electrodes, the melt may become contaminated with free silicon which is produced by reduction at a temperature below that of fusion (1800°). This is prevented by the use of perforated tubular electrodes which enable the vapour to be removed, by a tube of molten silica round the electrode, or by the presence in the sand of about 2% of water to oxidise the silicon vapour to silica. Except from the thermal point of view, the last method produces a superior product. J. Grant.

Prolonging the life of refractory material. W. Škola (Z. angew. Chem., 1927, **40**, 406—408).—Material for refractories should be burnt more than once, otherwise it is porous and incapable of withstanding heat, even if the temperature of burning is high (15—16 Seger). The refractory material is in a metastable condition after burning which changes to a stable condition during use, the change being a function of temperature and of the influence of furnace gases. Shrinkage and vitrification can be prevented during this change, and the life of the refractory prolonged by coating its surface with a special cement. Many such cements have been used comprising carborundum, rare earths, zinc oxide, and silicates, but good results have been obtained with "Resistin." It is applicable to the glass industry, chemical processes, and to oil or coal-dust fired furnaces. Illustrations are given of crucibles used for glass melting with and without a coating of the compound. W. G. Carey.

Removing iron from clay, kaolin, etc. H. Fleissner (Austr. P. 104,745, 17.12.24).—The material is calcined until it assumes a reddish tinge before it is subjected to the usual treatment for the removal of iron, *e.g.*, successive sulphurisation and extraction with acids. L. A. Coles.

Refractory lining. R. B. Pogue, Assr. to American Brake Shoe & Foundry Co. (U.S.P. 1,629,047, 17.5.27. Appl., 6.6.25).—The lining consists of about 40% of ganister, about 40% of mica schist, and 10—20% of fine clay, in such proportions that expansion and contraction of the ingredients under heat are compensated. F. G. Crosse.

Electrically heated [tunnel] kiln (E.P. 270,035).—See XI.

IX.—BUILDING MATERIALS.

Setting of dihydrates of calcium sulphate. P. P. Budnikov (Z. angew. Chem., 1927, **40**, 408—409).—The rapid hydration of anhydrite with water and a catalyst (cf. B., 1925, 172) is due to the formation of unstable complex hydrates on the surface of the anhydrite which decompose giving $CaSO_4,2H_2O$. Fine grinding increases the affinity of the anhydrite for water, and an acid salt as catalyst necessitates less grinding than does a neutral salt. Crystallisation plays an important part in setting, and as the hydration increases more crystallisation is observable. A cement can be made without burning by grinding natural gypsum with a catalyst (at most 0·3%) so that it passes a 9000/cm.^{2} mesh. The highest strength obtained was 70 kg./cm.^{2} after 28 days, using 0·3% of sodium hydrogen sulphate as catalyst. Sand can be added to this cement, and it can carry up to 30% of anhydrite. W. G. Carey.

High-temperature whitewash. E. P. Arthur, W. B. Mitchener, and J. R. Withrow (Ind. Eng. Chem., 1927, **19**, 591).—Whitewash suitable for application to industrial furnaces to facilitate cleaning and to prevent the infiltration of cold air to regenerators is made by stirring finely-ground lime into 5 gals. of water to form a fine slurry, and adding slowly to this 5 lb. of sodium chloride, 5 lb. of plaster of Paris, and ½ pint of sodium silicate. The wash is used immediately on brickwork, in two coats. W. G. Carey.

Influence of iron content on mortar strength. H. W. Leavitt and J. W. Gowen (Proc. Nat. Acad. Sci.,

1927, **13**, 263—265).—The iron content of the sand used is an important factor in influencing the tensile strength of mortar, the data presented showing that there is a slight but consistent increase in the strength developed by mortars on the 28-day test as the iron content of the native sand increases from traces only to 0·007%. The iron content is apparently without influence on the compressive strength. J. S. CARTER.

PATENTS.

Manufacture of aluminous cements in the revolving furnace. U. B. VOISIN (E.P. 270,496, 19.5.26).—A finely-ground, intimate mixture of bauxite and lime, preferably arranged so that the hydraulic index is greater than 2, *e.g.*, 75% of bauxite and 25% of slaked fat lime, is heated by hot air or gases at a temperature well below the softening point for a relatively long period in a revolving furnace. The product on grinding forms a rapid-hardening aluminous cement. B. W. CLARKE.

Colouring cements, plasters, plastics, etc. D. DE ROS and F. BARTON (E.P. 269,974, 27.1.26).—A colloidal solution of a hydroxide or a basic oxychloride of a metal, preferably iron, with the addition of a suitable pigment, *e.g.*, dehydrated oxide of iron, is added to cement, plaster, etc., producing colours ranging from yellow to terra-cotta. B. W. CLARKE.

Making concrete and products thereof. G. O. CASE (E.P. 270,013, 4.2.26).—Fibrous material, *e.g.*, sawdust, is impregnated with a dilute solution of calcium chloride, and mixed in the proportion of 2½—6 pts. by wt. with 1 pt. of dry cement, sufficient water being added to give the mixture the requisite consistency for use in making artificial stone, concrete, and the like. B. W. CLARKE.

Manufacture of bituminous concrete. L. S. VAN WESTRUM (E.P. 269,975, 27.1.26).—A bituminous soap, made by mixing bitumen with a saponifiable oil, which is subsequently saponified with alkali lye, is mixed with a mineral aggregate and dry cement, lime, or the like. B. W. CLARKE.

Production of highly-polished surfaces, especially on artificial stone. E. PROSIG (Austr. P. 104,738, 6.8.24).—A mixture of Portland cement or other binding agent, colouring material, and water is ground to such a degree of fineness that it possesses a glassy or silky lustre, and is then applied to the surface of the stone by brushing it on direct, or by pressing a thin layer of it on sheet metal on to the stone. L. A. COLES.

Linings for vessels to resist acids. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 260,575, 15.10.26. Conv., 27.10.25. Addn. to E.P. 258,616; B., 1927, 333).—Acid-proof plates, slabs, or the like, having a high thermal conductivity, for lining metal vessels, are prepared from powdered silicon added to water-glass cements described in the main patent. B. W. CLARKE.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Technical and economical conditions of the heavy metallurgical industry of the east of France, with particular reference to the utilisation of gases and motive power. J. SEIGLE (Iron and Steel Inst., May, 1927. Advance copy, 72 pp.).—The available ore is phosphoric, and the gangue either siliceous or calcareous; thus at many of the blast-furnaces the two types of ore can be mixed, so obviating the necessity of adding limestone. The ores are sent raw to the blast-furnaces. The bulk of the pig iron produced is made into either basic Bessemer or open-hearth steel, or used for castings. During the war, in order to increase the production of pig iron, steel turnings and scrap were put through the blast-furnaces; the steel thus becomes carburised during its descent through the furnace with the coke, and cast iron is produced. By this means the output of the furnace can be increased by 50% and the consumption of coke is decreased. The basic pig iron so formed is deficient in phosphorus, and this is compensated for by the addition of basic slag. The existence of fundamental relationships between the proportions of the constituents of the throat gases, carbon monoxide and dioxide, and the volumes of the blast and of the gas produced is demonstrated. Methods in actual use for improving the efficiency of the Cowper stove, for heating the various types of furnace, and for employing surplus blast-furnace gases are described. The means employed for power generation are described, and the efficiency of gas engines is discussed. It is shown that the theoretical consumption of steam per effective H.P. is much improved when the pressure is increased, and technical advances which take advantage of this are indicated. The steam is now superheated at 350—375°, and the effective steam-pressure on the turbines has steadily increased to 35 atm. The feed-water and the air for combustion of the blast-furnace gases are also heated, and various devices are employed to ensure an intimate and rapid mixture of the air with the gas for combustion. The basic process gives naturally a steel low in carbon; the addition of 0·6% of ferromanganese gives a steel with a tensile strength of 26 tons/sq. in. If extra soft steel is required, the after-blow is increased, and rather less ferromanganese added. For semi-hard or hard steels, recarburisation is adopted. The resistance of rails to wear is increased without introducing fragility, either by cooling the head of the rail by jets of air and water spray (Sandberg process), or by intermittently quenching it in a predetermined amount of cold water. A two-stage process for the manufacture of hæmatite pig iron from phosphoric pig iron is referred to in which the phosphoric pig iron is first dephosphorised in a converter, which results in a partial decarburisation, and then recarburised to the desired carbon content. Details of the dry-quenching of coke by the Sulzer process are given. M. E. NOTTAGE.

Interactions of gases and ore in the blast-furnace. I. At temperatures up to 650°. W. A. BONE, L. REEVE, and H. L. SAUNDERS (Iron and Steel Inst., May, 1927. Advance copy, 41 pp.).—The interaction of hæmatite ore and gases approximating in constitution to blast-furnace gas has been studied at varying temperatures between 380° and 650° by the "circulation method" introduced by Bone and Wheeler (Trans. Chem. Soc., 1903, **83**, 1074—1087). At all temperatures used, nearly all the carbonic oxide in the dry system was used up in ore reduction up to about 10% deoxidation, at which

point ore reduction was decelerated and carbon was deposited. At the highest temperature (650°) no carbon deposition supervened until 90% ore reduction had been effected. The introduction of 2% of hydrogen to the gases materially increased the proportion of carbon monoxide used in reducing ore and diminished the carbon deposition at 550°. C. A. KING.

Gaseous cementation of iron and steel. I. Cementation by carbon monoxide. A. BRAMLEY and A. J. JINKINGS (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 17—69).—The conditions necessary in order to obtain uniformity of results in gaseous carburisation are defined. In the constancy of other factors, the amount and distribution of carbon introduced by carbon monoxide carburisation depend on (i) the rate of flow of gas, (ii) period of carburisation, (iii) temperature, and (iv) initial concentration of carbon in steel. Rate of flow, whilst greatly affecting the weight of carbon introduced, has little effect on the depth of penetration, whereas both factors are influenced by period of carburisation and temperature. The higher the initial concentration of carbon in the steel, the lower is the amount of carbon introduced by cementation. No evidence of the liquation phenomenon observed by Giolitti was found. In all cases specimens were found to increase in diameter during carburisation. L. M. CLARK.

Gaseous cementation of iron and steel. II. Cementation with pyridine and methyl cyanide. A. BRAMLEY and G. H. BEEBY (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 71—125).—When pyridine and acetonitrile vapours are used as cementing agents, no advantage is gained by increasing the rate of flow of the cementing mixtures used beyond a certain value. Depth of penetration is a function of the period of cementation. With acetonitrile efficiency of cementation increases progressively up to the highest temperature investigated (1100°), whereas pyridine becomes much less efficient above 1000°. Mixtures of pyridine and carbon monoxide and of acetonitrile and carbon monoxide were also studied.
L. M. CLARK.

Diffusion of carbon and nitrogen into iron and steel. I. Diffusion of carbon. A. BRAMLEY and A. J. JINKINGS (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 127—153).—No variation in the diffusivity constant of carbon in iron and steel is caused by alteration of the period of carburisation, rate of flow of the carburising gas, or initial % of carbon in the steel, provided that the temperature is kept constant. Variation of temperature, however, has a very marked effect on the diffusivity constant. The relationship is given by the expression $\log K = 0 \cdot 00702t - 5 \cdot 730$, deduced from the experimental values established. L. M. CLARK.

Diffusion of carbon and nitrogen into iron and steel. II. Diffusion of carbon and nitrogen. A. BRAMLEY (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 155—174).—The diffusivity constants for carbon have been calculated from results of cementations with the vapours of pyridine and acetonitrile, and have been found to be substantially the same as those given in the previous paper. Nitrogen diffuses into iron and steel in a manner very similar to carbon, and obeys the same laws. The diffusivity constants for nitrogen for a number of temperatures have been calculated. The weight of the evidence deducible from the diffusivity constants for nitrogen and carbon, together with observations on the distribution of carbon in steel by X-ray methods, is strongly in favour of the solid theory of diffusion. L. M. CLARK.

Effect of constitution on the malleability of steel at high temperatures. O. W. ELLIS (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 195—215).—Pure iron forged in the neighbourhood of the A3 point is very much more malleable than material obtained at temperatures above or below this point. The forging temperature–malleability curve of a 0·9% carbon steel has a marked inflexion at about 865° whilst the malleability–temperature curve of any steel, whatever its carbon content, follows a linear law for temperatures above about 1000°. The malleability of a 0·11% carbon steel increases with great rapidity when a temperature of 1400° is passed. This alteration is ascribed not to any change in character of the iron, but to a change in the mode of distribution of the carbon in the solid solution. In the case of steels containing 0·46, 0·6, 0·8, and 0·9% C respectively, the malleability–temperature curve is strictly linear between 1000° and the solidus.
L. M. CLARK.

Dilatation of cast irons during repeated heating and cooling between 15° and 600°. R. HIGGINS (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 217—232).—Dilatation curves were taken of two series of cast irons containing approximately 1·5% Si. In the first series the phosphorus was low and the manganese varied; in the second the manganese was low and the phosphorus varied. The number of heats was limited to 21, the maximum temperature being 600°. The specimens were soaked at this temperature for 3 hrs. A small partial oxidation of the iron and graphite occurs. In the manganese series, the growth is due to oxidation and graphitisation, and an increase of the manganese content stabilises the iron against changes in the coefficient of expansion. In the phosphorus series increase in the phosphorus delays the growth, the phosphorus protecting the iron from oxidation. Phosphorus also increases the uniformity of the coefficient of expansion. Grey irons may show no growth after a certain number of heats, but their coefficient of expansion may be very erratic. L. M. CLARK.

Growth of commercial grey cast iron. C. E. PEARSON (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 281—317).—The conclusion of Rugar and Carpenter (cf. B., 1909, 1038) that growth, as ordinarily experienced, is primarily caused by oxidation of silicon and iron, and is greater the higher the silicon content of the iron, is confirmed. The rate at which iron cools after casting influences its susceptibility to growth. Iron heated under oxidising conditions reaches a maximum growth for any one heating within a few hours. Growth in the absence of oxidising conditions is almost negligible. The slight growth which occurs on repeated heating *in vacuo* appears to be due to the occluded gases in the iron.
L. M. CLARK.

Deformation of iron, with particular reference to single crystals. L. B. Pfeil (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 319—380).—A method is described for growing large crystals in ½ in. diameter iron rods. Finely-crystalline material and large crystals are employed in a study of the nature and mode of formation of Neumann lamellæ, produced by impact. The orientation of a crystal in relation to the direction of impact is unimportant. The force of impact determines the position of the lamellæ both in single crystals and in finely-crystalline specimens. After slow deformation, the lamellæ can no longer be produced by shock, but low-temperature annealing removes the effect of the deformation. The formation of a Neumann lamella produces a step on the polished surface of a crystal, and a step may also be produced at the intersection of two lamellæ. The lamellæ are not destroyed by prolonged annealing at high temperatures. It is concluded that Neumann lamellæ are twin crystals, and atomic movements are suggested which would produce twin lamellæ. Iron crystals with Neumann lamellæ in eleven out of the twelve possible directions have been obtained. Compression tests on single iron crystals are dealt with. It is shown that slip in an iron crystal does not occur on cubic or rhombododecahedral planes but on eicosetetrahedral planes. Very large crystals grown in iron strips tend towards one orientation, and the orientations of the crystals grown in round bars are considered in the light of this fact. L. M. Clark.

Influence of manganese on the properties of white-heart malleable cast iron. E. R. Taylor (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 381—406).—A manganese to sulphur ratio of 1·7 : 1 gives the best all-round results in a white-heart malleable cast iron, whatever the sulphur content may be. With this ratio, the lower the sulphur is, the better are the mechanical results. When the sulphur exists in the iron as ferrous sulphide, as much as 50% may be eliminated during annealing, but with the manganese-sulphur ratio of 1·7:1, or higher, sulphur is gained during annealing. Manganese in greater quantity confers strength without ductility. L. M. Clark.

Ac1 range in special steels. J. H. Andrew and H. A. Dickie (Iron and Steel Inst., May, 1927. Advance copy, 35 pp.).—From observation of the sp. vol. and hardness of a series of nickel steels, variation of the carbon content has no effect either on the position or extent of the Ac1 range, which depends on the nickel content of the steel. The temperature at which solution in γ-iron commences is lowered rapidly by an increase in nickel, and the amount of redistribution taking place in γ-iron depends on the quantity of nickel originally present in solid solution in the ferrite. Manganese gives an indication of effects similar to nickel, but of about 40% intensity; little effect is attributed to chromium. C. A. King.

Pseudo-twinning in ferrite, and solubility of carbon in α-iron at the A1 point. S. Tamura (Iron and Steel Inst., May, 1927. Advance copy, 7 pp.).—γ-Iron and austenite when annealed after being strained readily form twins; α-iron and ferrite do not. Twins in ferrite have, however, been observed recently in a sample of wrought iron and in another of mild steel. The latter was heated first at 680° for 18 hrs., then at 900 for 6 hrs., and finally cooled slowly in the furnace, when minute twin like crystals were found in the ferrite crystals. This twin-like structure was found to persist in both specimens after they were heated at 690° the one for 2 days and the other for 4 days. This apparent twinning also appeared in the new ferrite crystals after recrystallisation had been brought about by severely straining the specimen and then heating it at 690° for 18 hrs. As the formation of annealing twins is closely related to the atomic structure of the metal, and metals which, like ferrite, crystallise in the body-centred cubic lattice seldom produce them, these structures are not definitely stated to be true annealing twins, but are preferably called pseudo-twins. The solubility of carbon in α-iron is discussed on the basis of the fact that both α- and δ-iron have the same crystal lattice, and on the assumption that the point of maximum solubility of carbon in δ-iron lies on the solubility curve of carbon in α-iron, when this curve is extrapolated under specified conditions. On this assumption, the solubility of carbon in α-iron at the A1 point is 0·034% (cf. Yamada; B., 1927, 278). M. E. Nottage.

Cast iron with high manganese content from smelting manganiferous iron ores by the Siemens–Martin process. A. W. Smith (Iron Age, 1926, **118**, 1193—1194; Chem. Zentr., 1927, I., 514—515).—Manganiferous iron ores may be advantageously smelted to obtain a good cast iron when the manganese content does not exceed about 2%. With an ore containing 2% Mn the consumption of fluorspar in smelting is about 12% less than that required for an ore with 1% Mn. A. R. Powell.

Properties of some nickel-chromium-molybdenum steels. J. H. Andrew, M. S. Fisher, and J. M. Robertson (Iron and Steel Inst., May, 1927. Advance copy, 28 pp.).—The thermal and mechanical properties of two series of steels containing 1·6% Cr, 0·87% Mo, 2—5% Ni, and 0·4 (0·8)% C have been examined in some detail. The best combination of mechanical properties was obtained from the steels containing 0·4% C tempered at 625° for 3 hrs.; tensile strength 67 tons/sq. in., elongation 21%, and Izod value 39 ft.-lb. It is indicated that 650° may be within the critical range of high-nickel alloys. C. A. King.

Manganese and its properties: production of ferromanganese and its history. R. Hadfield (Iron and Steel Inst., May, 1927. Advance copy, 77 pp.).—The history of manganese is developed from its isolation by Gahn in 1774 to the present day, when over 400,000 tons are used in steel-making annually. The distribution, production, and average analysis of the manganese ore deposits in Brazil, Georgia, India, and the Gold Coast are discussed, and a map is given showing the ore resources of the world. None of the chief steel-producing countries is self-supporting in its manganese requirements, and the conservation of ferromanganese by the substitution of spiegel and high-manganese pig iron has been recommended. Manganese ores are chiefly used in the production of crude alloys in the manufacture of steel, also for making dry-batteries,

and in the glass industry. The m.p. of manganese is 1240°, and the sp. heat up to 1300° is given in a chart. Present-day manufacture in the blast furnace and electric furnace is described. The chief factor as regards purity is the phosphoric acid content in the ore rather than the process of production. Typical analyses of the blast-furnace products and of special products made in this country are given, together with the production capacity. 80% ferromanganese with 0·6—0·9% C is now being obtained. T. H. Burnham.

Non-rusting steel for flying machines. T. W. Downes (Iron Age, 1926, **118**, 1265—1268; Chem. Zentr., 1927, I., 518).—For the manufacture of tanks for aeroplanes etc., a steel of the following composition has proved satisfactory: 12·03% Cr, 0·45% Mn, 0·10% C, 0·26% P, 0·03% S, and 0·02% Si, and the remainder iron. A. R. Powell.

Ferroxyl indicator in corrosion research with especial reference to the problem of local corrosion. U. R. Evans (Metal Ind., 1926, **29**, 481—482, 507—508; Chem. Zentr., 1927, I., 518—519).—The usual ferroxyl indicator comprising a solution of potassium ferricyanide and phenolphthalein in agar-agar is liable to give misleading results when used for the detection of anode and cathode areas owing to the ferricyanide acting as cathode depolarising agent instead of oxygen. This difficulty is overcome by using the minimum amount of a solution made by mixing equal volumes of a 1% alcoholic phenolphthalein solution with a 1% aqueous solution of potassium ferricyanide. The cause of local pitting of iron may be due to the presence of particles of iron scale or of minute capillary openings in the surface of the metal (cf. Speller; B., 1925, 505). In moving water some of the particles of corrosion product formed in the early stages may become detached from the metal and be slowly washed along the surface, thereby giving rise to streaks of corrosion products. The conflicting theories of McKay (B., 1925, 136) and Liebreich (B., 1926, 57) are discussed in the light of the author's own work on the subject. A. R. Powell.

Determination of small quantities of copper in steel. J. D. Armour (Chemist-Analyst, 1925, [45], 3—5; Chem. Zentr., 1927, I., 151).—5 g. of steel are dissolved in 150 c.c. of 1 : 5 sulphuric acid, the solution is nearly neutralised with ammonia and saturated with hydrogen sulphide, and the precipitate collected, washed, and ignited in a silica crucible. The residue is fused with potassium pyrosulphate and the fused mass dissolved in hot water; 10 c.c. of an exactly 0·002% solution of copper as sulphate are added, and the total copper is determined iodometrically, allowance being subsequently made for the added copper. Before titration 20 c.c. of a 10% solution of potassium fluoride are added to prevent interference by traces of iron which are generally present. A. R. Powell.

Determinations of specific gravity of iron and low-carbon steel in a molten condition. D. W. Berlin (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 1—15).—Improvements in the apparatus devised by Benedicks, Berlin, and Phragmen (*ibid.*, 1924, **13**, 129) are described. The sp. gr. of four irons and steels of different carbon content are determined. Estimated mean values for different temperatures, of a preliminary character, are given. L. M. Clark.

Effect of annealing in steps on the specific electrical resistance of hard-drawn wires [of iron, nickel, and nickel alloys]. W. Rohn (Z. Metallk., 1927, **19**, 196—199).—The specific electrical resistance of hard-drawn iron and nickel wires increases in a smooth curve with rise of annealing temperature up to the respective transformation points, 860° and 360° respectively, at which points the curves become much less steep. In each case the change in the resistance is completely reversible except for nickel between 600° and 800°, when the resistance is slightly greater during heating than during cooling. The resistance of constantan wires (45% Ni, 55% Cu) at 20° decreases after annealing at 200—400° by up to 6% according to the annealing temperature, and a further 1% reduction takes place after annealing at 500—600°. Subsequent annealing at higher temperatures has no further effect on the cold resistance, but the resistance-temperature curve shows the same hysteretic anomaly at 500—700° as is shown by nickel at 600—800°. The step-wise annealing of nichrome wires (15% Cr, 22% Fe, 62% Ni, and 1% Mn) results in an increase in the cold resistance with an annealing temperature up to 500°. Higher temperatures up to 1000° have no further effect, but, after annealing at 1100°, another slight increase takes place, making a total increase of 9% over the resistance of the hard-drawn wire; at the same time the temperature coefficient of resistance decreases by 33%. A. R. Powell.

Commercial production of carbon-free chromium or ferrochrome by leaching from the ore and electrolysis. W. Crofts (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 175—194).—Chromite, roasted with lampblack carbon at 1350°, is reduced to oxides which can be leached from the gangue with concentrated sulphuric acid. After oxidation by aeration, iron can be fractionally precipitated by addition of calcium carbonate. The resulting chromium sulphate solution is electrolysed in a two-compartment cell, after the addition of sodium fluoride and magnesium sulphate. The chromium concentration is kept high by addition of chromium hydroxide, the p_H of the solution being kept at about 2·5. Carbon-free ferrochrome is obtained by electrolysis of the solution without removal of iron, the alloy appearing to contain the same proportion of constituents as was present in the solution. 1 lb. of chromium is deposited in 8 kw.-hrs. L. M. Clark.

Chromium steels for ball-bearings or rolls. V. Prever and G. Prever (Notiziaro Chimico-Industriale, 1926, **1**, 240—245, 272—276, 302—307, 346—355; Chem. Zentr., 1927, I., 349).—The best composition of chromium steels for use as ball-bearings and the like is comprised within the following range: 0·05—0·6% C, 11—16% Cr, 0—1% Mn, 0—2% Si, 0—25% Ni, 0—3% Mo, and 0—1·5% Cu. The higher the chromium content the better is the resistance towards organic acids and nitric acid, but not towards hydrochloric or sulphuric acids, the resistance to which is improved only by increasing the nickel content. A small proportion of copper tends to protect the alloy from

atmospheric corrosion, but has a deleterious effect on its working properties when hot. Molybdenum increases the cost of the alloy, but is most valuable in improving its acid-resisting properties, whilst silicon and manganese improve the working properties. A. R. POWELL.

Determination of phosphorus and silicon in ferrophosphorus. W. F. POND (Chemist-Analyst, 1925, [45], 16—17; Chem. Zentr., 1927, I., 150—151).—For the determination of phosphorus 0·2 g. is dissolved in 15 c.c. of nitric acid (*d* 1·4), 10 c.c. of hydrochloric acid (*d* 1·19), and 5 c.c. of strong sulphuric acid, and the solution evaporated until copious fumes of the latter are evolved, heating being continued for a further 1 hr. After cooling, 15 c.c. of nitric acid are added and the procedure is repeated. The solution is diluted with 50 c.c. of 1 : 1 nitric acid, boiled, and filtered, the filtrate being treated with ammonium molybdate as usual. For the determination of silicon, about 1 g. of the sample is dissolved as above, but heating with the strong sulphuric acid is continued for 2 hrs. The residue is dissolved in 25 c.c. of hydrochloric acid, 2 c.c. of nitric acid, and 125 c.c. of water, and the silica collected and determined in the usual way.
A. R. POWELL.

Burnt lime and raw limestone in the basic open-hearth process. C. H. HERTY, JUN. (Ind. Eng. Chem., 1927, **19**, 592—594).—The use of a mixture of limestone and burnt lime does not give such good results in open-hearth furnaces as when the materials are used separately. Burnt lime decreases the heat period, but it is somewhat restricted to charges low in pig iron where agitation is not necessary. With high pig iron charges the temperature of the bath during the early part of the heat is low, and limestone is advisable because the carbon dioxide evolved agitates the bath and allows the heat of the furnace gases to penetrate into the metal, the agitation also assisting the elimination of impurities in the steel. In burnt lime, sulphur should be low (0·05—0·10%), silica 2% or less, carbon dioxide and magnesia also should be low, a high magnesia content giving a viscous slag.
W. G. CAREY.

Structure and surface formation of cast copper. P. SIEBE and L. KATTERBACH (Z. Metallk., 1927, **19**, 177—186).—The casting temperature, mould temperature, and rate of casting are the chief factors which determine the macro-structure of copper cast in an open mould. The finest structure is obtained by pouring the metal slowly at about 1120° into a mould preheated at about 100°; a higher mould or casting temperature results in a coarse structure consisting of long, broad, needle-like crystals perpendicular to the walls of the mould and an elevated surface to the ingot. Too low a casting temperature or too rapid a rate of casting results in an uneven structure consisting of fine radial crystals on the outside of the ingot and coarse polygonal crystals in the interior. In the refining of copper by the poling process, "tough-pitch" copper gives the finest structure on casting; underpoled or overpoled metal invariably has a more or less coarse structure, accompanied by either a concave or convex surface to the ingot according to the conditions of casting. Lead, bismuth, and other impurities which do not form solid solutions with copper generally decrease the grain-size, but increase the tendency to piping, whereas metals, such as nickel, which form solid solutions with copper have the converse effect. A. R. POWELL.

Irregularities in silver[-copper alloys] and [methods of obtaining] silver [castings] free from blowholes. S. STREICHER (Z. Metallk., 1927, **19**, 205—210).—The chief faults to be met with in castings of silver–copper alloys containing 80—93·5% Ag are small blowholes or blisters, scaly patches which develop on rolling, and blue films which appear in the final polishing. All these are due to absorption of oxygen during melting. Part or all of this oxygen is converted into cuprous oxide, the amount of which increases with the duration of melting. If the cuprous oxide in the casting exceeds 1% of the total copper content of the alloy, the surface of the metal scales during rolling and small cracks appear during forging. Melting under a layer of carbon is not sufficient to prevent absorption of oxygen. The most efficient deoxidiser is 10% phosphor-copper, 1—3 g. of which are required per kg. of alloy made from fine silver and electrolytic copper, or 5—10 g. per kg. of scrap metal remelted. Cupromanganese and cuprosilicon may be used when a small amount of manganese or silicon is required in the alloy for improving its rolling properties. In order to obtain a homogeneous fine-grained structure, the castings should be forged with a light hammer (50 kg. from a height of 28 cm.), annealed at 600°, then flattened into sheet with a heavier blow, and finally rolled and polished. The usual enamels for silver melt at about 700°, at which temperature the grain-growth of worked silver takes place; to avoid splintering of the enamel after cooling the heating must be prolonged for a sufficient time to allow the grain-growth to proceed to completion.
A. R. POWELL.

Micro-structure of zinc coatings. W. H. FINKELDEY (Proc. Amer. Soc. Testing Materials, 1926, **26**, II., 304—316).—Metallographic specimens of zinc-coated materials should be polished after mounting in solid or fused zinc. Chromic acid is preferred for etching. Of the three layers, the inner two are iron–zinc alloys; it is, however, doubtful whether $FeZn_3$ is always present as well as $FeZn_7$. Thick or irregular alloy layers, or the presence of cadmium or lead in the zinc bath, caused poor bending properties.
CHEMICAL ABSTRACTS.

Age-hardening aluminium alloys. Substitution of beryllium for silicon in duralumin, lautal, and aludur. W. KROLL (Metall u. Erz, 1926, **23**, 613—616; Chem. Zentr., 1927, I., 515—516).—The limit of solid solubility of beryllium in aluminium is 0·3% at 640°, but this value is increased by the addition of magnesium; in both cases the solubility decreases with fall in temperature, hence the alloys are amenable to the age-hardening treatment. Thus the alloy containing 4·5% Cu, 0·6% Be, 0·6% Mg, and 0·1% Si has a tensile strength of 76 kg./mm.² after quenching from 520°, which increases to 106 kg./mm.² after keeping

at 20° for some days. The addition of beryllium to alloys of the aludur type causes a very marked improvement in their tensile properties; with lautal alloys, however, only a slight improvement is noticeable, and with duralumin practically none at all. The methods of determining magnesium and beryllium in aluminium alloys are briefly discussed. A. R. Powell.

Mechanical properties of binary aluminium–beryllium alloys. W. Kroll (Metall u. Erz, 1926, **23**, 616—618; Chem. Zentr., 1927, I., 516).—Addition of beryllium to aluminium increases the tensile strength, the alloy with 6% Be having a strength of 13·5 kg./mm.^{2} With up to 6% Be the elongation decreases slightly, but with further addition of beryllium decreases rapidly without any appreciable increase taking place in the tensile strength. The general effect of beryllium on the mechanical properties of aluminium is similar to that of silicon, but 30% more beryllium is required to produce identical results. A. R. Powell.

Indentation hardness of metals. K. Honda and K. Takahasi (Iron and Steel Inst., May, 1927. Advance copy, 11 pp.).—The authors' previous suggestion (B., 1924, 521) that the Brinell hardness number should be deduced from the diameter of the impression measured while the load is applied, and not after the removal of the load, is discussed. The method is now practically possible by the use of depth gauges fitted to the testing machine. The two methods are compared by experiments with copper, brass, and steels. With soft metals the one method may give larger or smaller results than the other, according to the nature of the material; with steels, the hardness numbers calculated from measurements made after the withdrawal of the load are always the greater. These differences are due to elastic recoil, and to the piling up of metal round the indentation. The authors' method gives results which are more simply related to those from scleroscope, impact, or pendulum testers than are those obtained by the usual method. W. Hume-Rothery.

Relation between the Brinell hardness and tensile strength of pure aluminium and aluminium alloys that undergo age-hardening. H. Bohner (Z. Metallk., 1927, **19**, 211—214).—The relation between the Brinell hardness and tensile strength in kg./mm.^{2} of sheets of duralumin, lautal, scleron, and aeron is given by the equation $T = 0{\cdot}343H + 4{\cdot}8$, provided that the metal has been reduced at least 90% in thickness by forging or rolling, and that it has subsequently been annealed, annealed and quenched, or annealed, quenched, and age-hardened. The corresponding relation for 98/99% Al is $T = 0{\cdot}34H + 1{\cdot}4$. The hardness test should be made with a ball of 2·5 mm. diameter with a load of 15·6 kg. for sheets 1·25—2·75 mm. in thickness, with a ball of 5 mm. diameter with a load of 62·5 kg. for sheets 2·5—6 mm. in thickness, and with a ball of 10 mm. diameter with a load of 250 kg. for thicker sheets. A. R. Powell.

Transformations undergone by aluminium bronzes. J. Bouldoires (Compt. rend., 1927, **184**, 1071—1073).—The dilatometric thermal analysis of the alloy previously described (B., 1926, 983) showed an anomaly on the heating curve at 325°, which became more pronounced after subsequent annealings. In air as medium the cooling curve showed a change of direction at 500°, but when cooling was accelerated by compressed air a marked anomaly with liberation of heat was obtained at 125—150°. This was due to the lowering of the temperature of the transformation recorded on the heating curve. After tempering in air the structure was martensitic. The results of the thermo-electric analysis were analogous to those obtained for the electrical resistance. J. Grant.

Coating of aluminium, magnesium, and light and ultra-light alloys. J. Cournot, J. Bary, and E. Perot (Compt. rend., 1927, **184**, 1172—1174; cf. B., 1926, 1018).—The penetration of aluminium by copper during the cementation process is not influenced by a current density below 0·7 amp.; above this a non-adherent deposit is obtained. Rapid cementation is also produced in a bath containing equal parts of sodium nitrite and potassium nitrate at low temperatures. Satisfactory cementation is not produced on pure aluminium with ferrochromes, nor on magnesium and its copper alloys, chiefly on account of the difficulty of producing efficient cleaning. Rapid and complete cementation of duralumin by ferrochromes of low carbon content was obtained after 1 hr. at 580°. A structure analogous to the Al_2Cu–Sol.η eutectic was observed, and the hardness was not increased. J. Grant.

Action of [various salt] solutions on aluminium and its alloys and on some rust-protecting materials. Samuel (Mitt. Kali-Forschungsanst., 1923, 103—105; Chem. Zentr., 1927, I., 518).—The action of 23—25% solutions of sodium and potassium chlorides and of magnesium sulphate on age-hardened duralumin, copper–aluminium (No. 12) alloy, and silumin, and on annealed and hard-rolled aluminium sheet has been investigated. At the ordinary temperature the reaction commences almost immediately with the formation of hydrogen bubbles and a voluminous white precipitate. After several days pronounced etching is visible in the case of duralumin and the copper–aluminium alloy, but the percentage loss in weight is very small. The most satisfactory protective coatings for iron or steel vessels to contain the above salt solutions comprise chromol–litharge and chromol–graphite pastes and aluminium sprayed on by the Schoop process, but none of these is resistant to hot (50—70°) solutions. A. R. Powell.

Blast-furnace slags containing titanium. A. Stansfield and J. E. Morrison (Trans. Roy. Soc. Canada, 1926, [iii], **20**, III., 439—443).—The softening points of mixtures of silica, lime, and titanium oxide (Ti_3O_5), with 10% of alumina when heated in an atmosphere of carbon dioxide and nitrogen, have been determined, and a triangular diagram is constructed. Mixtures containing 20—50% of silica, with 20—40% of lime, 10—50% of the titanium oxide, and 10% of alumina all soften below 1250°. At 1400° and above some nitride or similar compound of titanium seems to be formed, and tends to separate from the slag owing to its being more refractory. There is some danger of the formation of this compound in the blast furnace when working at high temperatures. Mixtures of silica, lime, titanium dioxide, and 10% of alumina heated in an oxidising atmosphere have

softening points 30—50° higher than the corresponding mixtures containing the oxide Ti_3O_5, but the diagram is otherwise very similar. The temperature of complete fusion appears to be 40—100° above the softening point.
R. CUTHILL.

Hardness and its relation to the cold-working and machining properties of metals. I. H. O'NEILL (Iron Steel Inst., Carnegie Schol. Mem., 1926, **15**, 233—279).—The existing data concerning static indentation tests are reviewed, and experiments correlating different sections of the subject are described. Elasticity effects in the indenting material and in the specimens are considered, together with permanent deformation of steel balls. Work was also carried out on diamond indenters in the form of spheres, pyramids, and cones. The hardness of annealed and cold-worked metals, as well as the flow of soft metals, are studied, and a scale of loading suitable for all metals is suggested.
L. M. CLARK.

Determination of the temperature at which a metal commences to recrystallise. G. TAMMANN and W. SALGE (Z. Metallk., 1927, **19**, 187—188).—If two highly-polished metal plates are laid one on top of the other on a horizontal plane, and the lower one is then slowly lifted, the angle of inclination to the horizontal at which the top plate slips off the other is a constant independent of the temperature up to the point at which recrystallisation commences. At this point the surfaces of the plates become slightly rough owing to the re-orientation of the crystals, and consequently the friction is much increased and the angle of slip also increases. The recrystallisation temperature may also be determined by observing when a highly polished surface of the metal begins to diffuse a beam of reflected light owing to the roughening that takes place. Using these methods, the temperature at which recrystallisation takes place in copper is 160—200°, in iron 170—185°, and in nickel 200—220°.
A. R. POWELL.

Electrosherardising. (Metal Ind. [New York], 1926, **24**, 459—461; Chem. Zentr., 1927, I., 517).—In commencing the electrosherardising process with new zinc material four or five charges of iron articles of low value should first be treated in order to obtain a satisfactory sherardising mixture (85—90% Zn and 8—10% ZnO). The lead content of this mixture should not exceed 1·5% or its m.p. will be too low for satisfactory work; more than 2% Fe increases the tendency to corrosion of the coatings. The accumulation of iron in the mixture is prevented by periodical magnetic separation.
A. R. POWELL.

Effect of metals on water supplies. CLARK.—See XXIII.

PATENTS.

Manufacture of basic open-hearth steel. J. L. SCHUELER (U.S.P. 1,628,281, 10.5.27. Appl., 27.3.26).—Basic open-hearth steel may be made from cold charges by first adding an insufficient amount of limestone to form a finishing slag with respect to the weight and kind of metal charged, and, after the charge has melted and without removing the slag formed, making up the insufficiency of limestone by the addition of burnt lime to form a finishing slag.
M. E. NOTTAGE.

Shaft furnace for reducing iron ores. Y. MURAKAMI (E.P. 269,959, 19.1.26).—In the throat of a blast furnace a vented hollow partition closed at the top diverts the descending charge towards valves on each side, by which the descent of the charge may be regulated. The valves conveniently are segmental cylindrical shutters having the convex surface uppermost and mounted on horizontal spindles for rotation.
C. A. KING.

Production of a strong, smooth, and pliable zinc coating on iron wire by a continuous process. MIDLAND MANUF. CO. (G.P. 436,694, 2.7.22).—The wire is passed continuously through a bath of molten zinc at 455—510°, then through a furnace maintained at 100—200° higher than the bath, the time of passage through the furnace being approximately three times that through the bath.
A. R. POWELL.

[Steel] alloy. F. F. LUCAS, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,628,990, 17.5.27. Appl., 8.10.25).—A steel alloy contains 7—20% Ni, 7—10% of a tungsten-like metal, 6—8% Cr, 2—7% Mn, and 0·5—0·6% C.
F. G. CROSSE.

Bearing metal of high lead content with a bronze basis. STELLA A.-G., and M. DREIFUSS (G.P. 436,440, 20.10.23).—The bronze used consists of phosphor-copper with one or more of the following elements: tin, zinc, vanadium, chromium, molybdenum, tungsten, or uranium. It is melted and poured into a molten lead alloy containing some copper and a little manganese. The resulting bearing metal is characterised by great toughness.
A. R. POWELL.

Alloys for making non-shrinking moulds. C. ZEISS (G.P. 435,917, 8.8.23).—Alloys suitable for making moulds of an exact shape and volume comprise at least 40% Bi with tin, lead, and cadmium. The following three alloys have a particularly small contraction on solidification: (*a*) 60% Bi, 15% Sn, 25% Pb; (*b*) 50% Bi, 43% Pb, 7% Cd, and (*c*) 44% Bi, 14% Sn, 24% Pb, and 13% Cd.
A. R. POWELL.

Lead alloys of easily oxidisable metals. CHEM. FABR. KALK G.M.B.H., E. HERRMUTH, and H. OEHME (G.P. 436,438, 24.11.23).—The readily oxidisable metals are placed in a container and covered with a sieve, on to which lead or a lead alloy is poured, at such a temperature that no alloying takes place until the sieve is completely covered with molten lead. The whole is then heated externally until alloying takes place.
A. R. POWELL.

Production of a metallic coating [*e.g.*, tin] on metal wires. V. PLANER (G.P. 437,502, 2.10.23).—The cleaned wires are passed through an emulsion of the powdered coating metal in an organic liquid, *e.g.*, tin dust in glycerin or oil, and subsequently heated to remove the liquid and cause the coating metal to adhere firmly.
A. R. POWELL.

Refining calcium and magnesium and their alloys. A. E. WHITE. From AMERICAN MAGNESIUM CORP. (E.P. 270,060, 24.3.26).—Calcium, magnesium, or their alloys are vaporised rapidly at an absolute pressure of not more than about 2 mm. of mercury. The metallic vapours may be condensed on a removable

liner composed of two hemicylindrical sections or on a surface having a temperature gradient ranging from above the sublimation point of the more volatile metal to a suitably lower temperature. C. A. King.

Protection of magnesium and its alloys from atmospheric oxidation. I. G. Farbenind. A.-G., Assees. of O. H. Weber (G.P. 435,931, 25.6.25).—By treating the surface of magnesium or of its alloys with a fluoride, corrosion of the metal by air or water is almost completely prevented. A. R. Powell.

Protection of metal articles [against attack by molten metals of low m.p.]. Siemens & Halske A.-G., Assees. of G. Masing (G.P. 436,092, 20.3.25).—The surface of the metal to be protected is coated with a substance which becomes soft at the temperature of the molten metal. Thus iron crucibles are coated internally with a mixture of powdered glass and alumina as a protection against molten aluminium. A. R. Powell.

Coating aluminium surfaces. E. H. Hewitson, Assr. to Eastman Kodak Co. (U.S.P. 1,627,900, 10.5.27. Appl., 23.8.26).—The surface, by treatment with a solution of an alkali zincate, becomes coated with zinc. The addition of a protective colloid, *e.g.*, starch, to the solution increases the density of the zinc. M. E. Nottage.

Extraction of chromium ore. Zahn & Co. Bau Chem. Fabr. G.m.b.H., and L. Wiekop (E.P. 270,143, 30.8.26).—The residue obtained after heating chromium ore together with an insufficient quantity of alkali carbonate, and extracting the heated mass, is again roasted with the theoretical quantity of alkali carbonate together with the addition of 2—3 pts. of lime to each part of iron oxide in the residue. As an intermediate step, undecomposed chrome ore may be separated from the lighter iron oxide and gangue, and recalcined with alkali carbonate. C. A. King.

Improving the properties of hard alloys, *e.g.*, stellite and akrite. Deutsch-Luxemburgische Bergwerks- u. Hütten-A.-G., and F. Bauerfeld(G.P. 436,439, 14.6.24).—The hardness, toughness, and cutting properties of hard alloys of the stellite type are improved considerably by melting the alloy in an atmosphere of nitrogen. A. R. Powell.

Decomposition of oxidised ores [tantalite, wolfram, etc.]. F. L. Hahn, and Firma W. Franke (G.P. 437,561, 14.8.25).—Ores consisting of tantalates niobates, tungstates, molybdates, or vanadates are decomposed by heating under pressure with carbon tetrachloride at 250—300°, and the products treated subsequently with dilute acid. A. R. Powell.

Silver alloy. R. H. Leach, Assr. to Handy & Harmen (U.S.P. 1,628,673, 17.5.27. Appl., 25.8.23).—The alloy contains silver together with 1—7% Cd and 6·5—0·5% Sb, the combined cadmium and antimony being 7·5%. F. G. Crosse.

Device for transforming the crystalline structure of [tungsten] wires. F. Koref, Assr. to General Electric Co. (U.S.P. 1,629,345, 17.5.27. Appl., 6.7.22. Conv., 13.7.21).—The wire is fed continuously across contacts of very high f.p. material, such as tungsten or molybdenum, the portion between the contacts being heated to high incandescence by an electric current supplied through the contacts (cf. Koref; B., 1923, 230 A). H. Holmes.

Concentrator. E. H. Johnson and A. H. Winterton (U.S.P. 1,627,648, 10.5.27. Appl., 21.10.25. Conv., 22.10.24).—A rotatable drum is arranged to rotate in one direction, the material to be treated being passed into the drum at one end and discharged at the other. The internal surface of the drum has riffles inclined inwardly and upwardly on the rising side of it at the intake end, and is provided with means to collect the concentrate from the surface. M. E. Nottage.

Apparatus for refining metals. M. Unger, Assr. to General Electric Co. (U.S.P. 1,628,375, 10.5.27. Appl., 31.12.25).—A crucible for molten metal comprises refractory material forming a reservoir for the charge and an annular channel in which a portion of the charge forms a closed secondary. Two openings spaced in the refractory material at right angles to the axes of the channel connect one end of the channel with the reservoir, whilst the opposite end is connected by a single opening. A primary winding is arranged to induce heating currents in the secondary and to circulate the molten metal by electromagnetic repulsion. J. S. G. Thomas.

Drying electric bright-annealing furnaces. Siemens-Schuckertwerke G.m.b.H. (E.P. 262,766, 3.12.26. Conv., 10.12.25).—In electric bright-annealing furnaces which operate with hydrogen as the protective gas, the gas is withdrawn by means of a rotary pump, purified from any vapours, *e.g.*, steam, formed in the furnace, and returned. C. A. King.

Electrolytic refining of copper containing tin. Siemens & Halske A.-G., Assees. of G. Hänsel (G.P. 436,084, 21.7.25).—During the electrolytic refining of copper containing tin the electrolyte becomes contaminated with colloidal stannic hydroxide. A portion is therefore continuously removed, treated with active charcoal to flocculate the tin compound, filtered, and returned to the circuit. The precipitate is treated in the usual way for the recovery of tin. A. R. Powell.

Chromium plating. Metropolitan-Vickers Electrical Co., Ltd., Assees. of J. D. Alley (E.P. 258,242, 26.8.26. Conv., 12.9.25).—Chromium is deposited from a solution containing 150 g. of CrO_3 and 4—8 g. of SO_4 per litre, the bath being maintained at 20—30°. The electrodes are separated by a distance of 0·5—1·5 in., a current density of 0·3—0·6 amp./sq. in. being used. C. A. King.

Production of chromium-plated articles with mirror-like, "coloured," or the like surface. Chromium Corp. of America, Assees. of C. G. Fink (E.P. 258,594, 15.9.26. Conv., 19.9.25).—A bright layer of chromium is deposited on a prepared, smooth, and highly polished foundation metal. The temperatures and current densities necessary for proper deposition are produced on a graph. C. A. King.

Electrolytic chromium. Olausson & Co. Aktiebolag (F.P. 612,880, 17.3.26).—For the deposition of chromium from an electrolyte consisting of a solution of chromic hydroxide in chromic acid, the soluble chromium anode is removed from the solution from time to

time and replaced by an insoluble anode; alternatively, a soluble chromium anode is used in parallel with an insoluble anode (*e.g.*, lead or mercury) or a partly soluble anode (*e.g.*, ferrochromium or other chromium alloy). An increase in the concentration of the electrolyte and a consequent high current density are thus avoided. A. R. Powell.

Galvanic bath. W. A. F. Pfanhauser, and Langbein-Pfanhauser-Werke A.-G. (E.P. 269,753, 3.6.26).

Manufacture of iron and steel. S. Westberg (U.S.P. 1,629,563, 24.5.27. Appl., 21.10.24. Conv., 14.4.24).—See E.P. 260,646; B., 1927, 46.

Sintering machine. Dwight & Lloyd Metallurgical Co., Assees. of J. Knox (E.P. 264,450, 9.9.26. Conv., 15.1.26).

Regenerative furnaces (U.S.P. 1,629,056 and E.P. 270,184).—See I.

Manufacture of copper sulphate (E.P. 264,510).—See VII.

Electrolytic separation and ore treatment (E.P. 269,749).—See XI.

XI.—ELECTROTECHNICS.

Application of potentiometric titrations in technical analysis. F. L. Hahn (with A. Krutsch, H. Déguisne, G. Weiler, and E. Hartleb) (Z. angew. Chem., 1927, **40**, 349—354).—The potentiometric method of titration offers important advantages over the usual method of determining end-points, particularly in technical analyses where the use of indicators is often hindered by the presence, in the sample liquid, of colour or suspended matter. A simplified arrangement of apparatus is described suitable for such work. The calomel electrode used has the side limb provided with two sintered glass discs, one at the junction and the other at the outlet, between which is a side tube connected through a tap to a reservoir of $0\cdot1N$-potassium chloride solution. This arrangement affords a convenient means for washing the limb between determinations and for renewing the buffer solution without disturbing the main portion of the cell. Examples are given showing the method of determining chromic salts directly by electrometric titration with potassium ferricyanide, of the halogens by means of permanganate, and of acids and bases. E. Holmes.

Electrical polarisation in selenium cells and the effects of desiccation. A. O. Rankine and J. W. Avery (Proc. Physical Soc., 1927, **39**, 187—202).—Measurements of the secondary *E.M.F.* displayed by selenium cells and the apparently abnormal effect of illumination on the corresponding secondary currents are described. Both are shown to be probably due to an invisible water film in parallel with the selenium. That this film is the principal seat of polarisation seems to be proved by the fact that polarisation disappears almost completely with prolonged drying. On this basis a satisfactory quantitative explanation of the various effects is given. An important practical consequence of the desiccation of the cell is the large increase obtained in its sensitivity to light. C. J. Smithells.

Determination of the power-voltage characteristic of a Siemens ozoniser. R. W. Lunt (Phil. Mag., 1927, [vii], **3**, 1025—1031).—An analysis of the factors involved in the method of measuring the power consumed in a Siemens ozoniser, described by Becker (B., 1921, 224 a), is given, together with a description of the appropriate technique. A. E. Mitchell.

Electrolytic chlorination. Jenks.—See XXIII.

Patents.

Electrolytic method and apparatus. K. E. Stuart, Assr. to Hooker Electro-Chemical Co. (U.S.P. 1,630,224, 24.5.27. Appl., 5.3.25).—In an electrolytic cell having longitudinally-arranged electrodes, gas evolved at an electrode is diverted along a submerged longitudinal path as so to produce uniform concentration of the electrolyte. J. S. G. Thomas.

Preventing the evaporation and oxidation of heated electrolytes. A. L. Mond. From F. Warlimont (E.P. 270,514, 22.6.26).—Alkaline electrolytes, *e.g.*, sodium sulphide, used in the electrolytic production or recovery of tin, are covered with unsaponifiable, preferably saturated, substances, *e.g.*, paraffin wax, which are liquid at, and have a b.p. considerably above, 80—110°. J. S. G. Thomas.

Electrolytic methods for separating and recovering the constituents of metallic salts in a state of fusion for use in the treatment of ores or other products. E. A. Ashcroft (E.P. 269,749, 27.11.25).—Sulphur or sulphur compounds are introduced into the fused electrolyte to combine with all the oxygen so that the destruction of carbon or graphite electrodes is prevented. If the electrolyte is a fused chloride, sulphur chlorides containing dissolved sulphur are produced which can be used for evolving or absorbing chlorine as may be necessary. When lead or zinc is to be produced the corresponding sulphide may be introduced. W. G. Carey.

[Electrolyte for] electrical primary or secondary cell. A. Cellino (E.P. 241,898, 19.10.25. Addn. to E.P. 225,197; B., 1925, 458).—Zinc ions are introduced electrochemically into an electrolyte containing silicic acid. J. S. G. Thomas.

Electrically heated [tunnel] kiln. B. J. Moore and A. J. Campbell (E.P. 270,035, 23.2.26).—The heating coils are hung freely on knife edges of refractory material, the circumference of the coils being greater than that of the coil supports. J. S. G. Thomas.

Induction electric furnaces. Ajax Electrothermic Corp., Assees. of E. F. Northrup (E.P. 243,694 and 247,519, 31.10.25. Conv., [a], 29.11.24, [b], 14.2.25).—(a) In an electric induction furnace the current frequency and diameter and resistivity of the charge are proportioned so that the induced electro-magnetic energy is substantially all converted into heat before reaching the axis of the charge. (b) In an electric induction furnace constructed in accordance with the previous patent, and intended for heating non-magnetic conducting materials, alternating current is supplied by a rotary multipolar generator of standard construction having power factor corrected by negative reactance. [Reference

is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907 and 1919, to [A] E.P. 218,967; (B., 1924, 794); 214,717; 119,220 and 111,844 (B., 1919, 375 A, 225 A); and 13,184 of 1908; also [in B] to E.P. 119,220 (B., 1919, 375 A).]

J. S. G. Thomas.

Electric furnace. I. R. Valentine, Assr. to General Electric Co. (U.S.P. 1,628,376, 10.5.27. Appl., 26.2.25).—An electric furnace comprises a refractory crucible having a duct in which the charge is heated by current passed through it, and a reservoir providing a head of molten charge communicating with the duct between its ends, whereby molten charge is supplied to the duct to prevent rupture of the electric circuit due to the pinch force. J. S. G. Thomas.

Manufacture of electric incandescence lamps. General Electric Co., Ltd., Assees. of Patent-Treuhand-Ges. für elektrische Glühlampen m.b.H. (E.P. 269,828, 9.2.27. Conv., 6.7.26).—An electron-emitting material, more especially colloidal thorium oxide, is mixed with the "getter" introduced into the lamp bulb to remove residual gases and prevent blackening. J. S. G. Thomas.

Metallic cores for electromagnets etc. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 269,770, 9.8.26).—The cores are made from fine metallic powders prepared by the thermal decomposition of metal carbonyls, *e.g.*, iron, nickel, or cobalt carbonyls, or mixtures thereof. J. S. G. Thomas.

[Conveying devices etc. for] galvanic mass production. Langbein-Pfanhauser-Werke A.-G. (E.P. 266,285, 18.9.26. Conv., 20.2.26).

Electrode carriers for electric furnaces. A.-G. Brown, Boveri, & Co. (E.P. 264,157, 4.1.27. Conv., 7.1.26).

Electric accumulator. E. Deodato (E.P. 256,569, 30.1.26. Conv., 7.8.25).

[Fixing terminals on electrodes of] primary voltaic cells. Comp. Franç. pour l'Exploit. des Proc. Thomson-Houston (E.P. 261,371, 8.11.26. Conv., 12.11.25).

Zinc sulphate solutions (E.P. 269,776).—See VII.

Colloidal silicic acid solutions (F.P. 612,486).—See VII.

XII.—FATS; OILS; WAXES.

Bleaching action of fuller's earth on oils. B. Neumann and S. Kober (Z. angew. Chem., 1927, **40**, 337—349).—The problem of the bleaching action of fuller's earth has been studied under three headings—the general phenomenon of bleaching, the effect of heat on fuller's earth, and the possible mechanism of the observed phenomena. The samples investigated were German proprietary products, and two specimens of American origin. The bleaching value of an earth was determined by a standardised method of agitating a given quantity of the earth with an oil, filtering, and examining colorimetrically the clarified filtrate. As colour standards, various vegetable oils and solutions of dyestuffs in mineral oils have been investigated. Vegetable oils are of little use, since variations in origin and treatment are reflected in subsequent bleaching, but 0·05% solutions of Fat-soluble Yellow AT, R, or 3R in paraffin oil are quite suitable. A complication may arise owing to the selective adsorption of the different earths for a particular dyestuff, but this difficulty is minimised if earths treated in the same way only are compared. It is noted that with vegetable oils, earths heated at 600° give the greatest bleaching effect, whilst in the case of the mineral oils containing dyestuffs untreated earths are the more effective. An explanation of this phenomenon is based on the assumption that the unheated earths have the greater surface area, and the fact that the dyestuffs used are crystalloids, whereas the vegetable oils contain appreciable quantities of colloidal matter, *e.g.*, mucilage and albumin, which may even act as colour carriers. It is supposed that as the size of the pore spaces increases with loss of water on heating, the resulting framework becomes more capable of retaining colloidal material, and less suitable for adsorbing the relatively small molecules of the dyestuff. The maximum bleaching of an oil is attained by using 2—4% by wt. of earth. In an attempt to correlate bleaching activity and chemical constitution the authors have determined the water loss, and the amounts of soluble aluminium oxide and iron oxide, in samples of earths heated for definite periods at temperatures up to 1000°, the soluble oxides being determined by heating 0·5 g. of the treated sample with 20 c.c. of 2*N*-hydrochloric acid under reflux for 1 hr. With five proprietary earths the water loss rose sharply to approximately 12% between 500° and 600°, whilst the soluble oxides reached a maximum between the same temperatures, but decreased rapidly between 800° and 1000°. In two cases the water loss rose steadily from 100° to 900°, and in one of them, *i.e.*, with the American earth, the soluble oxides were fairly constant at 8% each as high as 800°. These earths, with the exception of the American, gave the maximum bleaching effect on soya bean oil after treatment at 600°, this property increasing rapidly after heating at 100—300°, and falling again at 800°. The exception gave its maximum effect at 300°. It is concluded that the whole of the observed phenomena can be adequately explained on a physical adsorption basis.

E. Holmes.

Bleaching action of fuller's earth upon oils. G. Keppeler (Z. angew. Chem., 1927, **40**, 409).—The increased bleaching action of fuller's earth after heating at 400° (cf. Neumann and Kober; preceding abstract) is considered to be also due to organic constituents such as humus which are present in clays, although the amount may be so small that the colour is unaffected, and to the decomposition by heat of organic colloids and consequent freeing of the surface. Kaolin treated with alkali to dissolve humus bleaches more efficiently than when untreated. W. G. Carey.

Detection of linseed oil in soya bean oil. J. F. Carrière (Chem. Umschau, 1927, **34**, 113—120).—The hexabromide value of oils is determined as follows:—1—2 g. of the oil are neutralised, dissolved in 40 c.c. of ether and 5 c.c. of glacial acetic acid, and cooled to 0°, air being excluded. Bromine is added from a burette

until the coloration persists for 1 min., after which the coloration produced by 3 drops of bromine in excess should persist after keeping the mixture for 3 hrs. in ice water. The hexabromides are filtered, washed, and dried to constant weight at 100°. From a series of typical figures, limits and mean values for the relationships between the iodine value and hexabromide value of pure linseed oil and of pure soya bean oil are determined. For pure soya bean oil the value (iodine value — 126·19) ÷ hexabromide value is shown to be greater than 12, whilst for pure linseed oil (iodine value — 126·29) ÷ hexabromide value is approximately 1·574. Approximate and more accurate expressions for calculating the amount of linseed oil present in linseed oil–soya bean oil mixtures are deduced. These are uninfluenced by small quantities of other oils, with the exception of whale oil. S. S. Woolf.

Seeds of mercuriales. P. Gillot (Bull. Mat. Grasses, 1927, 59—62).—The seeds of *Mercurialis annua* (mercury), *M. perennis* (dog's mercury), and *M. tomentosa* yielded the following figures respectively on analysis: water 6·80, 8·58, 6·72; fatty matter 38·05, 26·45, 35·62; protein 13·55, 19·43, 15·25; cellulose 31·91, 39·55, 33·13; reduced sugars 0·09, 0·30, 0·07; hydrolysable sugars 1·29, 1·49, 0·97; starch 0·39, 0·10, —; ash 7·92, 4·10, 8·24%. Cold pressure or solvent extraction yields a limpid, odourless oil, of sweet taste and pale to golden-yellow colour. The oil varies but slightly for the three species, and has the following characteristics: d^{15} 0·934—0·937; n_D^{15} 1·4840—1·4861; iodine value 201·5—215·5; bromoglycerides 52—80%. It has a much higher iodine value than linseed oil, with which it compares very favourably as a drying oil, especially as it needs no preliminary purification. The chemical uniformity of the oil from the three species of mercuriales is the more remarkable on account of the distinct biological differences in the three plants. H. M. Langton.

Addition of iodine to unsaturated oils, fats, and fatty acids in an organic solvent. I. J. P. K. van der Steur (Rec. trav. chim., 1927, **46**, 278—283).—A 0·1*N*-solution of iodine in carbon tetrachloride was allowed to react with varied quantities of unsaturated oils, fats, and acids, kept in the dark; equilibrium was reached in 3 days. Equilibrium constants for the reaction —CH : CH— $+ I_2 \rightleftharpoons$ —CHI·CHI— are given for arachis oil, palm oil, soya bean oil, and the mixtures of acids obtained from these; for elaidic acid, and for oleic acid of varying degrees of purity; and for natural linoleic acid. In all cases except that of the purest oleic acid, the equilibrium constant increases with an increasing ratio of unsaturated compound to iodine; this is explained as being due to the co-existence of different equilibria in mixtures containing different unsaturated compounds. The oils have the same equilibrium constants as the acids to which they give rise; glyceride formation has, therefore, no effect upon the addition of iodine. E. W. Wignall.

Cresol–soap systems. I. Solubility of cresol soap solutions in water. S. Jenčič (Kolloid-Z., 1927, **42**, 69—79).—The solubility of cresol soap solutions in water depends both on the nature and the amount of the fatty acid in question. Of the sodium and potassium salts of the acetic acid series with even number of carbon atoms, the most readily soluble cresol soap solutions are provided by the laurates and myristates, which in themselves have colloidal properties and a ready solubility in water. The stearates and palmitates, which are comparatively slightly soluble, and the caprylates and caproates, which are crystalloidal, give feebly soluble creosol soaps. The alkali salts of unsaturated fatty acids of 18 and 22 carbon atoms, containing one or two double linkings, give cresol soaps of higher solubility than those obtained from saturated fatty acids with the same number of carbon atoms. The alkali salts of ricinoleic acid behaved similarly to the other unsaturated acids. E. S. Hedges.

Distillation of coconut oil at very low pressures. H. I. Waterman and J. A. Nijholt (Chem. Weekblad, 1927, 24, 268—269. Cf. B., 1926, 499).—An improved product is obtained by introducing a cooled receiver into the distillation flask, the usual exit tube thus serving to conduct away only the uncondensable gases. S. I. Levy.

Fat splitting and glycerin recovery. C. H. Keutgen (Chem.-Ztg., 1927, **51**, 62—64, 82—84).—In the autoclave method the ratio of the size of the charge to the content of the autoclave should be 1 kg. of fat to 2·2—2·3 litres, the vessel recommended for 2500 kg. of fat being 1·1—1·2 m. in diameter and 4·75—5·5 m. in length. Wrought iron covered with a strip of 4—5 mm. sheet copper, cast steel, cast iron, or copper may be used for autoclave construction. If dark fats be used in iron autoclaves the resulting fatty acids must be distilled, and copper or copper-lined vessels are preferable when light-coloured fatty acids are required. A copper autoclave 18—20 mm. thick regularly used day and night requires to be entirely renewed in 10—15 years, whereas a copper-lined vessel would require only the renewal of the copper lining in 7—9 years. Purification of the fats to remove albuminous and other foreign matter is achieved by appropriate filtration using siliceous earths, treatment with aluminium sulphate, etc., or by heating at 45—50° with ¾—1% of sulphuric acid (d 1·53—1·71), according to the nature of fat and its impurities. To the clear, purified fat 0·3—0·6% of zinc oxide, 0·1—0·2% of zinc dust, and 40—45% by wt. of water are added: the correct water ratio is important, for if this be less than 40% in general a higher pressure is required. With a steam pressure of 6—8 atm. and duration of 6—8 hrs. the degree of splitting is 85—92%, and with pressure 8—12 atm. for 8—9 hrs. the split may be 97—98%. The two-phase method of splitting for 6—7 hrs., removing the glycerin water, and, after addition of more water (but no splitting base), continuing for a further 3—4 hrs., is advantageous in some cases. In the Twitchell process in addition to the original reagent, the "Kontakt" and "Idrapid" have been used, the procedure being as with the old reagent. This process permits of 15,000 kg. of fat being split at each charge as against 2500 kg. by the autoclave method. The steam consumption is about the same by both methods. The Krebitz process depends on lime saponification in open vessels, about 25% of milk of lime being used and

the emulsion heated at 90—100° for 8—10 hrs. The enzyme process, though yielding beautifully clear fatty acids, gives a glycerin water more difficult to purify than that obtained by the other processes, owing to the presence of organic impurities, mostly compounds of nitrogen. The waters from the splitting, before evaporation for crude glycerin, undergo purification; those from the autoclave are neutralised with sulphuric acid, and then the addition of baryta water and aluminium sulphate causes impurities to be precipitated, whence, after neutralisation with barium carbonate, filtration follows. Where lime or magnesia is not the splitting base the glycerin water is made slightly basic with barium hydroxide before adding aluminium sulphate. The glycerin water is evaporated *in vacuo* to *d* 1·24. Twitchell glycerin water is similarly treated except that the first neutralisation is with barium carbonate and not sulphuric acid. A good crude glycerin should contain 88—89% of glycerol and, at most, 0·5% of ash and 1·0% of organic impurities. H. M. Langton.

Shea butter. G. de Belsunce (Bull. Mat. Grasses, 1926, 55—62, 195—198; 1927, 14—19).—Crude plantation butter contains 0·87% of insoluble matter, which when removed by filtration retains one tenth of the unsaponifiable and some colouring matter. Owing to the peculiar nature of the unsaponifiables, ordinary methods of determination give unreliable results. Full details of a special method are given, showing an average result of 3·63%. Treatment with boiling water removed the bulk of the odour, and this is further removed by steaming at not above 120° without vacuum. It is essential to deodorise before refining, which must be carried out in the first instance with sodium carbonate under the following conditions. Twice the theoretical sodium carbonate as a 35% solution at 95° is added to the oil at 95° kept agitated in an emulsifier. Cold brine is added after the neutralising and the temperature kept at 60—90°, when the soap stock separates at the top with a low refining loss. The free fatty acid is thus reduced to 3%, and the final refining is effected with the theoretical amount of caustic soda. The latter cannot be used in the first case as the refining losses are too heavy. After neutralising, a water-washing is essential to ensure stability. Bleaching is effected by adding 2% of alkaline persulphate to 2 pts. of oil and 1 pt. of water at 40—50°, without agitation. The mixture is slowly raised to the b.p. to complete bleaching, which is obtained without affecting the odour or taste. The defect of the oil for soap-making due to its unsaponifiable content can be overcome by oxidation with permanganate. Reduction in the unsaponifiable matter from 5·7% to 0·6% is recorded. This treatment is unsuitable for edible oils. A. Rayner.

Drying and thickening of fatty oils. Auer.—See XIII.

Patents.

Recovery of fats, oils, or oleaginous substances from fat-containing materials. W. T. Powling (E.P. 269,985, 29.1.26).—The nuisance caused by the free escape of malodorous vapours produced in fat-recovery operations is avoided if the melting ("degreasing"), subsequent extraction (with or without the use of a solvent), and fat-settling are all conducted in closed apparatus under conditions precluding the escape of gases etc. into the atmosphere. These gases are drawn through the system and are absorbed, or otherwise disposed of, on their final discharge. S. S. Woolf.

Dissolution or extraction of fats, oils, or allied materials. A. Eichengrün (E.P. 243,030, 17.11.25. Conv., 17.11.24).—Methylene chloride is used as extraction solvent alone or in conjunction with other solvents or non-solvents. S. S. Woolf.

Manufacture of soap. H. W. D. Schou (E.P. 266,291, 23.10.26. Conv., 20.2.26).—Suitable proportions of oils, fatty acids, etc. and lye (of 10—50% strength) are emulsified at temperatures up to 80°. On cooling, the solid or semi-solid stabilised emulsion is allowed to saponify spontaneously, being moulded and even packed before chemical action is completed. S. S. Woolf.

Preparation of plastic materials. M. Melamid (G.P. 434,143, 7.2.23).—The products described in G.P. 386,062 (B., 1924, 302), obtained by treating the distillation residues of fats, resins, etc. with substances containing sulphur, are mixed with indifferent materials such as asbestos; or they are mixed with acetylene condensation products, with or without the addition of indifferent substances, and the mixtures transformed into plastic masses by combined heat and pressure treatment. The products can be used for luting, or as insulating materials etc. A. B. Manning.

Rendering solids of animal origin containing fat and oil. Allbright-Nell Co., Assees. of W. Laabs (E.P. 253,952, 22.6.26. Conv., 22.6.25).—See U.S.P. 1,578,245; B., 1926, 449.

Manufacture of oleomargarine (E.P. 252,369).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Oxidation in the drying and thickening of fatty oils. L. Auer (Farben-Ztg., 1927, **32**, 1729—1731, 1790—1792).—Further discussion of the author's gas-coagulation theory (cf. B., 1926, 450). The observations on which the older oxidation theories rest are recapitulated, and shown to be compatible with the view that stand oil formation and film formation are two stages or varieties of one and the same colloidal process of aggregation common to all drying oils, whether of the tung, linseed, poppyseed, or olive oil type. The possibility of structural chemical changes subsequent to these primary changes in degree of dispersion is admitted, such changes being dependent on the composition of the oils and the various "resinophore" groups present. The oxygen in the so-called "oxy-acids" of Fahrion is adsorbed and activated, not chemically combined, support for this statement being furnished by the work of Gelissen and Hermans, Staudinger, and Szent-Györgyi on organic peroxides and activated oxygen, a summary of which is given. The influence of ionised gas on the degree of dispersion of the charged suspended oil particles is discussed, and such phenomena as the acceleration of the drying process by exposure to ultra-violet light etc. are considered from this point of view. The determination of the

"carbon number" (*i.e.*, percentage change in carbon content during the drying process) is of assistance in ascertaining the nature and amount of volatile decomposition products more exactly than heretofore, such information being still needed for the elucidation of the drying process. S. S. Woolf.

Plasticity and flow-determinations of paste paints. H. Vollmann (Farben-Ztg., 1927, **32**, 1904—1906).—Blanc fixe was shaken up with various liquids, and the sedimentation of the suspensions observed. With liquids of the "resinous" type, *e.g.*, crude turpentine, tetralin, linseed oil, a protective action is noted, leading to slow settling, close packing of the sediment, poorly marked line of division, and retention of colloidally-dispersed solid in the supernatant liquor. In the case of "non-colloidal" liquids, *e.g.*, benzene, freshly-distilled turpentine and tetralin, etc., a definite separation of a more loosely-packed sediment occurs. The influence of vehicle and pigment on the plasticity of paste paints is discussed. The rates of flow of pastes (1 pt. of pigment : 1 pt. of medium) of blanc fixe, zinc oxide, lithopone, and chrome yellow in linseed oil, stand oil, and an ester gum varnish were determined using a modified Gardner flowmeter. The linseed oil pastes are shown to possess relatively high plasticity, contrary to the ester gum varnish pastes. The stand oil results are less striking, considerations of viscosity etc. intervening, but low plasticity is indicated. S. S. Woolf.

Determination of colophony in shellac. M. Reinbeck (Farben-Ztg., 1927, **32**, 1793—1796).—Existing methods for determining rosin in shellac are reviewed, consideration being mainly given to the iodine value method and the light petroleum extraction method. These are shown to be untrustworthy when applied to bleached shellac, as, according to the various bleaching processes used, the presence of as much as 30—40% of rosin may be undetected. Physical tests, *e.g.*, softness of sample, tackiness when used as polish, etc., may give information, but this, too, is to be regarded with suspicion. S. S. Woolf.

Hard Manila copal. J. Scheiber (Annalen, 1927, **453**, 52—61).—The abnormally high acid value of the acid, m.p. 160—162°, obtained by H. Wolff (Farben-Ztg., 1925, **31**, 807) from hard Manila copal was probably due to adsorbed mineral acid. By a modified process 35% of a crystalline acid, m.p. 204°, which is identical in every respect with the principal acidic constituent of soft Manila copal, has now been isolated from the hard variety. The finely-divided mixture of acids obtained by dissolving the copal in 5% potassium hydroxide and reprecipitating with hydrochloric acid is extracted for some days with 5—10% ammonium carbonate solution. The crude acid, m.p. 100—150°, recovered from the filtrate attains after repeated recrystallisation the m.p. 204°, acid value 320·5, and iodine value 78·4. It corresponds in composition and mol. wt. (in phenol) with the formula $C_{18}H_{30}(CO_2H)_2$ or $C_{17}H_{28}(CO_2H)_2$, and gives colloidal solutions in many solvents. It is proposed that the term mancopalolic acid be retained for this substance, which is also present in pontianak resin, of which the α- and β-mancopalolic acids and mancopalic acid of Tschirch and Koch (A., 1902, i, 478), and Richmond's acid, m.p. 185—187° (*ibid.*, 1910, i, 690), were probably impure specimens. Hard Manila copal probably differs from the soft variety only in containing larger amounts of polymerised and autoxidised products. The residue of resin acids insoluble in ammonium carbonate contains an *acid*, m.p. about 280°, acid value 190. H. E. F. Notton.

Behaviour of phenolic resins. A. A. Drummond (J. Oil and Colour Chem. Assoc., 1927, **10**, 83—100. Cf. B., 1925, 365).—The effect of volatile matter in soluble phenolic resins on the physical properties, particularly solubility, of the resins is examined. A method of determining the weight lost by thin films of resins on heating at 60°, 110°, and 120°, successively, is described, the total loss attaining nearly 50% in the case of some commercial and laboratory-prepared resins tested. Pure resins were prepared in the soluble, fusible state, practically free from matter volatile at 90—100°, and their solubility in a number of alcohols and esters was determined, as well as the effect on their solubility of temperature changes and of added substances of solvent or of non-solvent nature. In the last case (the addition of water to butyl acetate solutions of a phenol–formaldehyde resin being taken as a typical example), the susceptibility of the equilibrium to disturbance is shown to vary with the composition, considerable ranges of miscibility being established. A series of photomicrographs of aged resin films illustrates the influence of small residues of free volatile matter. S. S. Woolf.

High-temperature whitewash. Arthur and others.—See IX.

Patents.

Production of silicate colours. Van Baerle & Co., Chem. Fabr. (G.P. 437,553, 14.6.25).—Potassium silicate containing 10—30% of water is used as binding agent. L. A. Coles.

Manufacture of condensation products of carbamide or a derivative thereof and formaldehyde. O. Y. Imray. From Soc. Chem. Ind. in Basle (E.P. 246,126, 4.1.26).—If the condensation of carbamide or a derivative thereof and formaldehyde or its polymerides be carried out under increased pressure and, if desired, at a temperature above 100°, the reaction is accelerated whilst the consistency of the product is under complete control. S. S. Woolf.

Manufacture of resin-like substances. Griffiths Brothers & Co., London, Ltd., and R. P. L. Britton (E.P. 269,973, 27.1.26).—1 mol. of a monohydric aromatic side-chain alcohol, *e.g.*, benzyl or cinnamyl alcohol etc., or a halogen or other derivative thereof is treated with not less than ⅓ mol. of a condensing agent, *e.g.*, sulphuric acid, phosphoric anhydride, etc., the temperature being regulated according to the nature and amounts of the reagents etc. The aromatic alcohol may be produced *in situ* from an aromatic hydrocarbon and formaldehyde in equimolecular proportions. The resin is isolated from the reaction mixture by extraction (five methods being detailed) and is purified by alkaline treatment. S. S. Woolf.

Production of compositions containing phenol-aldehyde condensation products. Bakelite Ges.m.b.H. (E.P. 247,957, 16.2.26. Conv., 17.2.25).—See G.P. 431,514; B., 1926, 889.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Patent.

Briquetting coal (F.P. 611,548).—See II.

XV.—LEATHER; GLUE.

Effect of filling materials on the strength and stretch of sole leather. D. Woodroffe and D. B. Gilbert (J. Soc. Leather Trades' Chem., 1927, 11, 68—75).—Samples of woven fabric were treated with myrobalans extract, quebracho extract, 10% gum tragasol, molten paraffin wax, concentrated gum tragasol, molten tallow, and sulphonated oil, respectively, then tested for tensile strength and stretch. Tanning extracts and sulphonated oil strengthened the fabric, whilst gum tragasol, tallow, and paraffin wax weakened it. The filling material appears to take the strain if it has less stretch than the fabric, and the fabric if it has not, which explains the increased strength with tanning materials and the weakening effect of gum tragasol. The stretch was less with the weaker treated fabrics. The tensile strengths and stretch of a number of vegetable-tanned insole leathers are quoted, and it is shown that the weaker leathers possessed greater stretch. It is suggested that the greater strength and less stretch are due to the filling of tanning extract. Some of the physical properties of vegetable-tanned sole leather appear to be due to the properties of the filling material.

D. Woodroffe.

Effect of a synthetic tan (" Gerbstoff F ") on the properties of some vegetable tannins. E. Stiasny and F. Orth (Collegium, 1927, 189—202).—Hot solutions of various tannin extracts were prepared of four times analytical strength, mixed with one eighth the amount of " Gerbstoff F," heated on the water-bath for ½ hr., diluted to analytical strength, cooled, and analysed. The insoluble matter diminished with quebracho, oakwood, and gambier extracts, but increased with mimosa, chestnut, and oak bark extracts. The non-tans increased in every case except that of sulphited quebracho by the synthetic tan addition. The different products were salted out with ⅓-, ⅔-, and fully-saturated salt solutions, and the amounts of the different fractions determined. The amount in quebracho and " Gerbstoff F " mixtures which could not be salted out was 5·5% greater than that in quebracho alone, which points to dispersion of the tannin particles. The portion salted out by ⅓-saturated salt solution was increased by 18·8%, due to the solubilising of insoluble matter, and similarly with oakwood extract. The portion of oakwood which could not be salted out was diminished by the " Gerbstoff F " treatment. Gambier showed a dispersion of all the particles which could be salted out. Chestnut showed a diminution in the amount of the coarser soluble particles, but an increase of all the others. With oak bark the first fraction was diminished, the second and third fractions increased, and the portion remaining was diminished by addition of " Gerbstoff F." Experiments with different amounts of synthetic tan and mimosa extract showed that a small amount of the former increased the size of the particles, whilst a larger amount caused dispersion. The addition of " Gerbstoff F " to the above extracts resulted in an increase in the Wilson–Kern tannin content of from 2% with oak bark to 120% with gambier. The speed of tannage was determined for the various mixtures; " Gerbstoff F " increased the intensity of tannage as measured by the amount of tannin irreversibly fixed.

D. Woodroffe.

Gelatin precipitation test for tannins. A. E. Jones (Analyst, 1927, **52**, 275—276).—A positive gelatin precipitation test is not a specific indication of the presence of a tannin. Positive tests were given by (in 1% solution) gallic acid, β-resorcylic acid, hæmatoxylin, brazilin, picric acid, ethyl gallate, *m*-hydroxybenzoic acid, hydroxyquinol, and maclurin; in saturated solutions, phenol, resorcinol, pyrocatechol, phloroglucinol, pyrogallol, methyl gallate, guaiacol, and protocatechuic acid, whilst gum arabic, starch, inulin, and catechin were amongst the substances giving negative results.

D. G. Hewer.

Patents.

Tanning process. Mathieson Alkali Works, Assees. of F. S. Low (E.P. 256,979, 12.8.26. Conv., 13.8.25).—Hides are exposed to the action of a solution containing basic chromium chloride formed by dissolving anhydrous chromous chloride in water under oxidising conditions, or by dissolving anhydrous chromic chloride with the aid of a reducing agent, *e.g.*, ferrochrome, or by using a mixture of anhydrous chromic chloride and a metal, *e.g.*, zinc, capable of reducing it.

D. Woodroffe.

Manufacture of chromium-magnesium preparation suitable for tanning purposes. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 255,087, 8.7.26. Conv., 10.7.25. Addn. to E.P. 251,267; B., 1927, 440).—Magnesium chromate or dichromate is reduced in presence or absence of an acid to produce a basic chromium-magnesium compound.

D. Woodroffe.

Manufacture of sulphurised derivatives of naphthols [tanning agents]. Fabr. van Chem. Producten, and E. Kraus (E.P. 269,971, 26.1.26).—Products useful as tanning agents are made by boiling a mixture of a naphthol, sulphur, and an alkali in water for 40—80 hrs., and treating the resulting mixture with formaldehyde or acetaldehyde and a sulphite. The alkali used must be equivalent, or preferably less than equivalent, to the naphthol, and in place of α- or β-naphthol a mixture of a naphthol and phenol or homologues of these may be employed. Thus β-naphthol (144 pts.), water (100 pts.), sodium hydroxide (20 pts.), and sulphur powder (80 pts.) are heated under reflux for 40—80 hrs. until a sample precipitated by acid is easily soluble in cold chloroform or carbon disulphide. The mass is then diluted with water (250 pts.) and crystalline sodium sulphite (250 pts.) and 40% formaldehyde (80 pts.) are added. A resin which separates redissolves on stirring for 2—3 hrs., and after keeping for 12 hrs. at 15° and heating for 1 hr. at 70—80° a clear solution of the new

tanning agent is obtained; the product may be salted out if desired. C. HOLLINS.

Production of diaphragms constructed of hardened gelatin or glue [for electro-osmotic use]. I. G. FARBENIND. A.-G., Assees. of J. REITSTÖTTER (G.P. 437,909, 17.1.24).—The diaphragms are hardened by treatment with polymerides of formaldehyde.
L. A. COLES.

XVI.—AGRICULTURE.

Effect of fertilisers on germination of seeds. J. L. MAXTON (Soil Sci., 1927, **23**, 335—341).—Seeds are not appreciably injured by remaining in contact with dry fertilisers for periods up to four weeks. All the fertilisers tested did, however, reduce germination after the seeds were sown in moist soil, the extent of the injury varying with the kind of seed and with different fertilisers. Calcium cyanamide caused the greatest injury. Seeds of a variety of agricultural crops, and a number of nitrogenous, phosphatic, and potassic fertilisers were used in the experiments. C. T. GIMINGHAM.

The hydrometer as a new means for the mechanical analysis of soils. G. J. BOUYOUCOS (Soil Sci., 1927, **23**, 343—353; cf. B., 1927, 422).—A method is proposed for the measurement, by means of a sensitive hydrometer, of the rate of settling of soil particles dispersed in water, and thus for obtaining a curve from which the distribution of the particles of various sizes can be calculated. The hydrometer reading gives an average of all the different densities in the column of liquid from top to bottom. 75 g. of soil are washed 2 or 3 times with distilled water to remove soluble salts, and rubbed gently with a pestle to break the compound particles. The soil is then mixed with an excess of water, allowed to remain a few seconds, and the supernatant liquid poured into a cylinder. This process is continued until all dispersable material is dispersed. The suspension is made up to 1075 c.c. in the cylinder and shaken vigorously for 5 min. Hydrometer readings are then taken every minute for as long as necessary; about 1 hr. is usually sufficient. C. T. GIMINGHAM.

Preparation of soil suspensions and degree of dispersion as measured by the Wiegner-Gessner apparatus. G. WIEGNER (Soil Sci., 1927, **23**, 377—390. Cf. Gessner; A., 1926, 377).—Studies are reported on the influence of the electrolytes in a soil suspension on the final result of shaking, rubbing, or boiling for 1 hr., as methods for the dispersion of the particles. The boiling process gives greater dispersion than shaking or rubbing, if the soil is first thoroughly washed, and has only a very small content of electrolytes, whereas in suspensions from the same soil not carefully washed, boiling produces stronger coagulation than the other treatments. Similarly, freezing a soil suspension tends to coagulate the particles if electrolytes are present, but if they are first removed, has no detectable influence on the degree of dispersion. With soils containing carbonates, the greatest degree of dispersion was obtained by treating first with dilute hydrochloric acid, then removing the electrolytes by thorough washing, and, finally, adding $0 \cdot 1N$-ammonia solution.
C. T. GIMINGHAM.

Factors affecting the hydrogen-ion concentration of soils. L. D. BAVER (Soil Sci., 1927, **23**, 399—414).—Determination of the p_H value of a soil should be made on an unground, fresh, moist sample. Grinding affects the reaction seriously; air-drying affects it only slightly, and air-dried samples can be used for most purposes. There is a periodical variation in the p_H of field soils during the year. Fertilisers, except ammonium sulphate, have little influence. The effect of liming extends to the subsoil. C. T. GIMINGHAM.

Decomposition of calcium cyanamide in the soil. M. VERMEIRE (Natuurwetensch. Tijds., 1927, **9**, 40—44). —A *résumé* of the observations and theories recorded in the literature. S. I. LEVY.

Rate of soil liming. J. A. SLIPHER (Ind. Eng. Chem., 1927, **19**, 561—564).—A study of 17 liming experiments to reduce acidity made upon United States soils shows that fractional rates of liming are more efficient per unit of lime than are full applications, the first increment of lime changing the p_H value of the soil more than each additional one, and the lightest rate of liming effects the greatest proportionate change in soil reaction. If the additions of lime are in arithmetical relation, the resultant soil reactions are in geometrical relation, and the crop response is in accordance with this, *i.e.*, light rates of liming produce proportionately more crop growth than heavy rates, and in geometrical relationship. This was found to be true for maize, wheat, oats, clover, barley, etc., although cereals respond to liming better than do legumes, but they are also more tolerant of acid soils. W. G. CAREY.

Importance of potash salts (and naturally-occurring impurities) for soils and for plants used for human food. O. NOLTE (Landw. Versuchs-Stat., 1927, **106**, 1—123).—The literature pertaining to the use of potash salts in plant production and the relationship between soil chemistry and plant physiology is comprehensively reviewed and discussed.
A. G. POLLARD.

Soil colloids and critical soil humidity for higher plants. K. FLEROV (Kolloid-Z., 1927, **42**, 66—69).— The maximal hygroscopicity was measured for ignited sand, soil, silica gel, alumina gel, manganese dioxide gel, and ferric oxide gel by desiccation and weighing. Comparisons were made of the growth of plants in each of these media, observation being made of the time of withering of the plant through lack of water. It was found that the individual gels have different available amounts of water for disposal to the plants. The "coefficients of withering" are given for silica gel 4·04, ferric oxide gel 9·24, and alumina gel 10·48. This coefficient has a very variable value for soils, as the different soils and different layers of the same soil have different contents of colloids, and the colloids themselves differ in properties. E. S. HEDGES.

Influence of carbon dioxide on sugar beet and other crops. L. ZALESKI (Mem. Inst. Nat. Polonais d'écon. rurale à Palawy, 1926, **7**, 29 pp.; Chem. Zentr., 1927, I, 957).—There is a definite increase in dry matter and sugar in sugar beet grown in an atmosphere rich in carbon dioxide. The increase in content of sugar was more marked in the leaves than in the roots.

The total amount of extractives in the leaves of the "carbon dioxide" plants was greater, and the total ash was less, than in plants grown in the open air. There was a slight, but constant, increase in the content of nitrogen in the leaves, and decrease in the roots. The effect of carbon dioxide on peas was sometimes beneficial, and sometimes harmful. Enrichment of the air of a hot-house with carbon dioxide trebled the total yield of dry matter of mustard. With buckwheat and millet, an initial advantage, due to additional carbon dioxide, was almost entirely lost, owing to the unfavourable conditions for growth in the hot-house.

C. T. Gimingham.

Patents.

Fungicidal and bactericidal agents. Ver. für chem. u. met. Prod. (G.P. 438,241, 18.6.24).—Aromatic or aliphatic aldehydes are condensed with quinaldine or pyridine derivatives, salts, or complex salts, and the products are used alone or mixed with other suitable fungicides or bactericides. Thus, infected seeds after being treated for an hour with a 1% solution of the condensation product from *m*-nitrobenzaldehyde and a quinaldine salt, or from cinnamaldehyde and dimethylaminoquinaldine ethiodide, or from dimethylaminobenzaldehyde and 2 : 4 : 6-trimethylpyridine ethiodide, or from glyoxal and ethoxyquinaldine ethiodide, are free from blight-spores, such as *Ustilago* or *Fusarium*. Ciliates, isolated from their host, are precipitated at once by treatment with a 0·05% solution of the condensation products, and amœbæ after a short time draw in their pseudopods, with formation of a cyst. C. Hollins.

Process of sprouting buds and plants. F. E. Denny, Assr. to Boyce Thompson Inst. for Plant Research, Inc. (U.S.P. 1,628,035, 10.5.27. Appl., 14.7.26).—The process consists in treating buds before planting with a halogen derivative of ethylene. *E.g.*, potatoes are immersed in a solution of ethylenechlorohydrin. C. T. Gimingham.

Carbonic acid for fertilising plants (G.P. 438,186). —See II.

XVII.—SUGARS; STARCHES; GUMS.

Lime problems in the beet sugar industry. R. W. Shafor (Ind. Eng. Chem., 1927, 19, 573—576).—Lime which can be readily and finely ground is necessary for the beet sugar industry. The calcium oxide in lime powder is not all effective for the Steffen reaction, the amount of reagent required being inversely proportional to the area per unit weight of lime. The area per unit weight can be measured by the percentage "free settling" of the lime in a series of inverted cones when subjected to a flow of paraffin oil from a constant-head feed, or it may be estimated with sufficient accuracy for commercial operations by finding the amount of lime reagent needed for a standardised sugar solution. W. G. Carey.

Patents.

Manufacture of white sugar. W. F. R. Murrie (U.S.P. 1,615,846, 1.2.27. Appl., 1.12.24).—After liming the raw juice, settling, and decanting, a suitable precipitant is added to throw down the lime and iron compounds. The juice is again settled and decanted, then concentrated, and the syrup obtained passed through filter-presses, being finally clarified by filtering over a carbonaceous material to remove soluble organic and inorganic matter prior to crystallising in vacuum pans.

J. P. Ogilvie.

Production of pure sugar liquors. C. F. Kullgren and S. G. Lind (U.S.P. 1,616,131, 1.2.27. Appl., 5.10.25. Conv., 13.5.25).—Sugar liquor is forced upwards from below through vessels containing a layer or layers of a purifying media consisting of silicic acid produced in the known way by treatment of an alkali silicate by an acid. J. P. Ogilvie.

Production of inulin and its derivatives. W. C. Arsem, Assr. to Industrial Technics Corp. (U.S.P. 1,616,164, 1.2.27. Appl., 1.10.26).—An impure solution containing inulin is treated with magnesium hydroxide, filtered, and the inulin precipitated by the addition of a water-soluble alcohol to the purified solution. J. P. Ogilvie.

Extraction and purification of inulin. W. C. Arsem, Assr. to Industrial Technics Corp. (U.S.P. 1,616,167, 1.2.27. Appl., 1.10.26).—Coagulation of impurities is effected by subjecting the juice to the action of a mixture of calcium hydroxide and calcium chloride, then decolorising, and removing further impurities by addition of an adsorbing decolorising agent, a solution of sodium carbonate being added to precipitate the calcium.

J. P. Ogilvie.

Hydrolysis of inulin. W. C. Arsem, Assr. to Industrial Technics Corp. (U.S.P. 1,616,169, 1.2.27. Appl., 12.7.21).—A solution containing inulin is subjected to the action of an acid in any desired concentration, the action being continued until the rotation of polarised light produced by the solution passes through a maximum, decreases, and again increases to a further and higher maximum than that first obtained. J. P. Ogilvie.

Hydrolysis of inulin. W. C. Arsem, Assr. to Industrial Technics Corp. (U.S.P. 1,616,171, 1.2.27. Appl., 14.8.24).—A solution containing purified inulin is subjected to the action of an inorganic acid in a concentration corresponding to about $0 \cdot 05N$ for a period of about 5 min. J. P. Ogilvie.

Production of lævulose. W. C. Arsem, Assr. to Industrial Technics Corp. (U.S.P. 1,616,165—6, 1.2.27. Appl., [a] 14.8.24, [b] 29.3.20).—(a) A solution containing purified inulin is treated with sufficient acid to produce a p_H approximately equivalent to $0 \cdot 01N$, the solution being kept at an elevated temperature until the desired conversion into lævulose is effected. (b) A basic substance is added to an acid aqueous extract containing inulin until it contains less than about 0·00001 g.-mol. of acid per litre, and not more than 0·00001 g.-mol. of dissolved base per litre, the mixture is then heated, and the precipitate collected.

J. P. Ogilvie.

Preparation of lævulose. W. C. Arsem. Assr. to Industrial Technics Corp. (U.S.P. 1,616,172, 1.2.27. Appl., 14.8.24).—Purified inulin is subjected to the action of less than about 70% of water and an amount of organic acid sufficient to produce a p_H corresponding to from 0·05 to $0 \cdot 015N$, this acidified solution being heated to convert it into lævulose.

J. P. Ogilvie.

Manufacture of a lævulose product. W. C. ARSEM, Assr. to INDUSTRIAL TECHNICS CORP. (U.S.P. 1,616,170, 1.2.27. Appl., 4.10.26).—Claim is made for a new pure food product comprising a colourless solution of lævulose and containing tartaric acid. J. P. OGILVIE.

Purification of sugar juices. W. C. ARSEM, Assr. to INDUSTRIAL TECHNICS CORP. (U.S.P. 1,616,168, 1.2.27. Appl., 16.5.21).—Impurities are precipitated by the action of a base more basic than calcium carbonate, and a normal alkaline-earth metal salt of an acid stronger than carbonic acid, which salt alone is capable of precipitating impurities from the impure solutions, the amount of both base and salt being sufficient to maintain the p_H between 6 and 8, and the amount of alkaline-earth metal ion being sufficient to react with all the precipitable impurities in the solution. J. P. OGILVIE.

Extraction of juice from sugar cane etc. F. MAXWELL (E.P. 263,893, 9.7.25).—Cane is subjected to cyclically-repeated groups of operations, each comprising crushing, agitating and/or shredding, and macerating steps, transposably associated in near or distant relationship with each other. The mass of crushed cane is subjected to agitation simultaneously, or substantially so, with the supply of the macerating agent at a position adjacent to or spaced from the region of initiating this agitation, which region may be anterior or posterior to the region of the supply of the macerating agent. Disaggregation may be applied to the crushed cane at high speed, and the macerating agent may be supplied in a dissipated state. J. P. OGILVIE.

Extraction of sugar from molasses. L. STEFFEN (E.P. 266,187, 11.6.26. Conv., 20.5.26).—Sugar is precipitated as an insoluble saccharate by treating dilute molasses with calcium oxide, the addition of which is stopped when there remains only about 0·5% of sugar in the solution. The tricalcium saccharate is worked up by filter-pressing, and then completely precipitated by the addition of an excess of lime. The insoluble saccharate thus finally obtained is separated by filter-pressing, mashed with a fresh dilute molasses solution, mixed with further dilute molasses, and the soluble sugar-lime produced is precipitated as insoluble saccharate with calcium oxide. J. P. OGILVIE.

Evaporator for sugar juices etc. A. VINCIK and F. TUREK (G.P. 427,780, 20.6.24).—In an evaporator of the type having heating devices arranged about a middle chamber divided into several divisions by separating walls, a circular tube is introduced into the lower system of connecting tubes, and this, by means of suitable confining elements in one of the heating devices, is divided into a corresponding number of sections. In the two systems separated from one another by the tube, confining elements are likewise provided, thus permitting the isolation of one or more heating devices from operation as needed. In the last division of the middle chamber a double-armed lever operated by a float is arranged, which closes the inlet or outlet valves at high or low level, and also permits the direction of flow of the valves to be reversed at high level. J. P. OGILVIE.

Extraction of cell juices. ÉTABL. BYLA (F.P. 606,932, 6.3.25).—Animal or vegetable matter cut into pieces is dispersed with saccharine substances which absorb the cell juices to form a liquid. The concentrated liquid is mixed with a certain amount of sugar, and the mass evaporated to dryness, to form a product containing proteins in finely-divided and water-soluble form. J. P. OGILVIE.

Treatment of sugar juices. M. J. H. SAVARY (F.P. 612,946, 19.3.26).—In order to avoid the darkening of the juice expressed from sugar-beet, sugar cane, etc. on exposure to air, a solution of monocalcium phosphate is added, either before extraction or pressing, or to the juice as it comes from the press. It may also be used in the diffusion process. C. T. GIMINGHAM.

Bagasse board (U.S.P. 1,627,103).—See V.

XVIII.—FERMENTATION INDUSTRIES.

Analyses of potable spirits made from wine and from vinasse, and of raw and rectified spirit. F. PIRANI (Annali Chim. Appl., 1927, **17**, 176—187).—Results are given of determinations (1) of the usual components in 190 samples of brandy made from Italian wine and vinasse, and (2) of the proportions of methyl alcohol in 17 samples of rectified spirits from wine and vinasse. In the determination of the acidity, use is made, not of phenolphthalein, but of tincture of litmus (cf. Barbet, Proc. VIth Internat. Congress Appl. Chem., 5, 645), the solubility being increased by diluting the spirit with boiled distilled water. In determining the esters, it is essential to effect the saponification with calcium saccharate (Barbet, *loc. cit.*, 648), which does not resinify the aldehydes, since otherwise the coloration developed obscures the colour change of the indicator. Even when kept away from the air, calcium saccharate solutions vary in titre, so that a blank test is necessary: 25 c.c. of the spirit are boiled for 2 hrs. in a reflux apparatus with 50 c.c. of about 0·1*N*-calcium saccharate solution, the quantity of 0·1*N*-sulphuric acid required to neutralise such 50 c.c. being added immediately after the boiling, and the excess of acid then measured by titration with 0·1*N*-alkali. Denigès' colorimetric determination of the formic acid produced on oxidation of the methyl alcohol present seems the best adapted for determining the methyl alcohol in spirits; the test is best made on 1 c.c. of the liquid, and the comparison tube should have the same alcoholic strength as that tested. Alcohol from the distillation of wine never contains more than 0·35 vol. of methyl alcohol per 100 vols. of anhydrous ethyl alcohol, whereas with vinasse spirit up to 4 vols. are found. In rectified spirits from wine and vinasse, the proportions are trace—0·25 and 0·54—1·90 respectively. T. H. POPE.

Total iron content of wines. J. H. FARBRE and E. BRÉMOND (Ann. Falsif., 1927, **20**, 213—217).—Ferrous salts are always present to the extent of about 10—14 mg./litre in wine manufactured from grapes pressed by treading and fermented and matured in wooden and glass vessels. An abnormally high iron content may produce ferric tannate, which imparts a bitter taste and a blue-black tint to the wine, rendering it unsaleable.

It is shown that dressing the vines with ferrous or ferric sulphate does not affect the iron content of the resultant wine. Wines from machine-pressed grapes, and which were fermented in ferro-concrete vats, were found to contain as much as 60 mg. of iron per litre and exhibited the "iron complaint" to a marked degree. The conclusion is drawn that the sulphur dioxide added to the wine during fermentation attacks the cast iron door fittings and the iron reinforcements of the walls of vats, by permeating the cement facings. The amount of iron thus introduced into the wine is sufficient to cause the spoiling of the wine. This could be avoided by the use of copper or enamelled fittings and glass storage vessels.

H. J. DOWDEN.

Influence of potassium hydrogen tartrate and of tartaric and malic acids on the precipitation of protein substances, in relation to the defecation and clarification of musts. E. ERRICHELLI (Annali Chim. Appl., 1927, **17**, 221—244).—The coagulation of proteins in presence of sufficient tannin to precipitate them is favoured by addition of tartaric acid up to about 0·005%, but hindered by larger proportions, 0·015% of the acid completely preventing precipitation. Malic acid and potassium hydrogen tartrate exert a similar influence. The bearings of these observations on the defecation of grape must and on the clarification of wine are discussed.

T. H. POPE.

Presence and detection of furfuraldehyde in vinegar. L. H. LAMPITT, E. B. HUGHES, and L. H. TRACE (Analyst, 1927, **52**, 260—265).—Vinegar gives rise to furfuraldehyde on heating at 80°, and in commercial distillation the amount of distillate increases as the process proceeds, and at the same time there is a reduction in the pentosan content. Furfuraldehyde may be determined in light-coloured vinegars by Youngburg and Pucher's method (cf. B., 1924, 1028), but for dark vinegars 6 c.c. of redistilled aniline are dissolved in 24 c.c. of glacial acetic acid and made up to 60 c.c. with pure amyl alcohol. To 10 c.c. of this mixture 20 c.c. of the vinegar are added, shaken, and allowed to remain in the dark for 15 min. The amyl alcohol which separates is deep red in colour if furfuraldehyde is present, 0·1 pt. of furfuraldehyde in a million being thus detectable.

D. G. HEWER.

Effect of pre-treatment on the properties of top-yeast and bottom-yeast. H. FINK and H. VON EULER (Z. physiol. Chem., 1927, **163**, 193—201; cf. von Euler, Fink, and Nilsson, A., 1926, 1176).—The removability of the co-enzymes in dry yeast preparations by lixiviation depends on the conditions of growth, especially on the composition of the nutrient material used. If bottom-yeast is cultivated in liquor containing sucrose, the co-enzymes can no longer be removed by washing; the same condition results in top-yeast grown in beer-wort. The capacity to hydrolyse raffinose is shown, on the other hand, to be independent of the conditions of growth or of the nutriment used, and does not run parallel with the removability of the co-enzymes by washing. Only after prolonged intensive culture is a top-yeast able to hydrolyse raffinose, and this is the result of much more profound change than the solubilising or insolubilising of the co-enzymes.

C. HOLLINS.

Rumanian alcohol. ZAHARIA and MOTZOC.—See XX.

PATENTS.

Manufacture of yeast. J. WEBER, Assee. of H. CLAASSEN (E.P. 264,795, 1.11.26. Conv., 21.1.26. Addn. to E.P. 230,098; B., 1925, 897).—Ammonium salts are added to a portion of the nutritive solution containing organic nitrogen, and the yeast fermentation is carried on until the greater portion of the inorganic nitrogen has been absorbed. The resulting yeast crop, with or without separation from the liquid medium, is then added to the remaining quantity of the original nutritive solution.

C. RANKEN.

Vitamin preparation (U.S.P. 1,624,154).—See XIX.

XIX.—FOODS.

Comparison of some physical and chemical tests for determining the quality of gluten in wheat and flour. D. A. COLEMAN, H. B. DIXON, and H. C. FELLOWS (J. Agric. Res., 1927, **34**, 241—264).—A study of the results obtained by several methods in use for determining gluten quality, and comparison of these with the baking data on the same samples of flour. The highest coefficient of correlation with baking strength, as shown by the loaf volume and crumb texture, was given by the crude protein content. Next in order ranked the washed gluten test, the viscosity determination of one concentration of flour and water, the kernel texture of the wheat, and the water-absorbing power of the flour. Determinations of the quality angle b (cf. Sharp and Gortner; B., 1924, 29), and of the viscosities of flour suspensions containing the same amount of protein were of no value as a measure of gluten quality, but the lack of agreement between these and the volume of the loaf was probably due in part to variations in baking procedure.

F. R. ENNOS.

Rapid determination of the [weight of] dry gluten. MARION (Ann. Falsif., 1927, **20**, 210—213).—The dry weight of gluten is determined by introducing a known weight (w) of the moist material into a small, wide-mouthed, stoppered bottle of known volume (V), and measuring the volume of water (E) now required to fill the bottle. The dry weight (W) is then given by the equation: $W = w - 0{\cdot}72\ (V - E)$. The figure 0·72 is an empirical factor derived from the observation that the volume of moist gluten is 3·56 times that of the dry material.

H. J. DOWDEN.

Biochemical method for detecting watering of milk. G. RIMINI (Annali Chim. Appl., 1927, **17**, 214—220).—The following test appears to indicate infallibly the addition of 10% or more of water to goat's or cow's milk. The milk is boiled for some minutes and allowed to cool to 50°, 50 c.c. being then introduced into a wide-mouthed phial, into which 1 c.c. of well-shaken, cold, acidified milk (yoghurt) is next pipetted. The vessel is closed with a ground stopper, thoroughly shaken for a few seconds, and immersed in a bath kept at 40—45° so that the surface of the milk is just beneath that of the bath. If the milk is genuine, a compact coagulum forms without separation of serum; if, however, 10% of water had been added to the milk, the coagulum obtained is soft and a shallow layer of whey appears

The time required for coagulation varies with the proportion of added water. Observation of the tube should commence 90 min. after addition of the acidifying organism, the tube being withdrawn from the bath and slightly inclined, being carefully replaced if the milk is still liquid. Further observations are made at intervals of 10 min., and when coagulation is noted, the tube is allowed to cool to room temperature and examined. Coagulation occurs rather more rapidly in watered than in genuine milk. Milk sold as "sterilised fresh milk" answers to this test, giving a compact coagulum without separated serum, but the resulting product exhibits no acid taste, so that in this case the coagulation is effected by the casease without the help of lactic acid.

T. H. POPE.

Use of lime in butter making. O. R. OVERMAN (Ind. Eng. Chem., 1927, **19**, 571—573).—Butter made from cream of high acidity develops an unpleasant flavour on keeping, and on pasteurising such cream the casein is coagulated. The use of such substances as sodium carbonate or bicarbonate to neutralise the acidity is convenient, and the carbon dioxide evolved is said to remove undesirable flavours and odours, but a soapy taste is imparted to the butter. Calcium carbonate is unsuitable owing to its insolubility, but in the United States the use of dry hydrated lime is extensive. It is applied as a suspension of 2 lb. of hydrated lime in 1 gal. of water, the cream being violently agitated and the lime water introduced as a fine spray. The acidity is reduced to about 0·3% and not below 0·2%, titrations being made with 0·1*N*-sodium hydroxide and phenolphthalein.

W. G. CAREY.

Apple juice in "pure fruit" jams. C. F. MUTTELET (Ann. Falsif., 1927, **20**, 208—210).—The detection by microscopical methods of the illegal addition of apples to so-called "pure fruit" jams, has been evaded by using filtered apple juice. It is shown that such addition can be proved, either by the presence of malic acid (cf. B., 1922, 726 A), or by the relative proportions of lævulose and dextrose present (cf. B., 1927, 122). In apple juice, lævulose always predominates, whilst in other fruits used for making jams the dextrose content either equals or exceeds that of lævulose. Application of the second method to the investigation of oranges and mandarins has shown that in 100 c.c. of juice, the dextrose exceeds the lævulose by 0·08—0·5 g. according to the variety. Home-made marmalade showed a dextrose excess of 3%, but two commercial samples containing, respectively, 1·75% and 2·25% excess of lævulose had clearly been adulterated with apple juice.

H. J. DOWDEN.

Occurrence of glass fragments in foods packed in glass containers. G. C. HANCOCK (Rep. Public Health and Medical Subjects, 1927, No. 37. 36 pp.).—The evidence accumulated shows that the occurrence of glass fragments of considerable size is rare, but not unknown. The source of such fragments may be in the manufacture of the container itself, especially in the case of hand-made bottles, or in the filling of the container with food. Glass particles of microscopic size are of frequent occurrence, but they are not necessarily caused by the containers, the dust in the air having been proved to be a fruitful source of such impurities. Glass particles may be distinguished from sand or other mineral matter by their appearance in polarised light. Under the polarising microscope with crossed nicols, glass particles are invisible, whilst crystalline particles appear bright on a dark field and often show interference colours. Amorphous quartz etc. has a mottled appearance.

H. J. DOWDEN.

Determination of benzoic acid in foodstuffs. G. W. MONIER-WILLIAMS (Rep. Public Health and Medical Subjects, 1927, No. 39. 57 pp.).—Methods for the determination of benzoic acid fall into two classes, (*a*) extraction by solvents and (*b*) steam distillation. To avoid the formation of emulsions in the extraction method, it is necessary to remove proteins by precipitation with acetic or phosphotungstic acids, or mercuric nitrate. The coagulum is liable to retain some benzoic acid. The precipitation may be performed by a saturated solution of sodium chloride, but in this case owing to the low solubility of benzoic acid in strong salt solution (0·08% in the saturated solution), free benzoic acid may be precipitated. Removal of proteins may also be effected by prolonged boiling with caustic alkali or hydrochloric acid. Benzoic acid may be extracted from materials containing fats by shaking with a weak solution of sodium bicarbonate, but if the fat is present in large quantities it should first be removed before extracting the benzoic acid. In methods of the second class it is established that, in the presence of saturated sodium chloride solution, benzoic acid is completely volatile in steam. Whether obtained by extraction or distillation, the benzoic acid is always impure and must be purified by washing with water, by oxidation with alkaline permanganate, by sublimation, or by precipitation of metallic benzoates, but each of these processes has its limitations. The most sensitive test for benzoic acid is that proposed by Mohler (cf. B., 1890, 770). The buff-coloured precipitate produced by addition of ferric salts to neutral solutions of benzoates is very delicate, as is also Jonescu's test (cf. B., 1909, 670). The method recommended by the author for the determination of benzoic acid consists in distilling the material in steam after saturation with sodium chloride and adding phosphoric acid. The distillate is evaporated to small bulk and impurities are oxidised by treatment with alkaline permanganate. The acid is then extracted by a mixture of equal volumes of methylated ether and light petroleum (b.p. 30—50°), after first saturating with sodium chloride. The extract is drawn over into specially-arranged test-tubes, and the solvent removed by aspirating air. The crude solid benzoic acid is purified by mixing with pure sand and subliming in a special oven at about 160°, a layer of filter paper preventing contamination of the sublimate by the sand. After cutting off the lower end of the tube containing the sand, the sublimate is weighed *in situ*.

H. J. DOWDEN.

Determination of sulphur dioxide in dried fruit. P. MAY (Analyst, 1927, **52**, 271—273).—100—200 g. of a fruit of the class in which 2000 pts. of sulphur dioxide per million are allowed, or 40 g. of moister fruits in which 750 pts. per million are allowed, are weighed into a flask containing 25 g. of marble and connected by a

splash-head to a condenser with its adapter dipping into 100 c.c. of saturated bromine water. 25 c.c. of concentrated hydrochloric acid diluted to 300 c.c. are added to the contents of the flask, and, after the evolution of carbon dioxide has slackened, the flask is heated until all the gas has been evolved, after which 200 c.c. are distilled over. The distillate is then evaporated to about 120 c.c. and the barium sulphate precipitated and weighed. Results were consistent, and likely sources of error tend to give low results. A considerable number of tests with unsulphured fruits gave consistent zero results. D. G. Hewer.

Formation of hydrocyanic acid from linseed meal, and its dependence on the hydrogen-ion concentration. P. Hansen (Dansk Tidsskr. Farm., 1927, 1, 382—388).—Linseed meal contains a glucoside which, in the presence of water, can be split up by enzyme action into sugar, acetone, and hydrocyanic acid; the amount of the acid formed may reach 0·4%, and many cases of cattle poisoning have been traced to this source. Collins and Blair (A., 1915, i, 110) showed that the production of hydrocyanic acid was hindered by the presence of quite small amounts of an acid. Experiments now show that the optimum temperature for the production of hydrocyanic acid from linseed meal lies between 40° and 50°, and, further, that the optimum hydrogen-ion concentration for the reaction lies about p_H 5. No liberation of hydrogen cyanide took place with p_H values greater than 8 or less than 2. The observed p_H values for suspensions of linseed meal, measured with a quinhydrone electrode using a Veibel normal electrode for comparison, exhibit a marked deviation from those calculated from the strength of the hydrochloric acid employed. The differences are probably due to adsorption effects, although buffer effects and the presence of ampholytic substances may also be responsible. H. F. Harwood.

Lime-treatment of pea cannery wastes. Warwick. —See XXIII.

Patents.

Production of bread. E. Rosenbaum (Swiss P. 115,100, 10.12.25).—After leaving bran in contact with water until fermentation begins, the liquid is poured off from the residue, worked up with flour into dough, and baked. F. R. Ennos.

Sterilisation, pasteurisation, or like treatment of milk and other liquids. R. L. Munday (E.P. 269,678, 25.1.26).—The liquid is exhausted of air by spraying or filming it or by agitating it in bulk within a vacuum chamber, whence it is taken by a positive-action vacuum pump to a series of heat exchangers, in which it is sterilised. Further, it may be subjected to the action of ultra-violet rays, or impregnated with sterile air or a flavouring or odorising gas, a continuous vacuum being sustained throughout the whole process. F. R. Ennos.

Making a self-preserving acid milk product especially adapted for the lower animals. A. P. Hunt (E.P. 269,610, 24.11.25).—Skimmed milk is treated with a suitable culture, *e.g.*, *B. Bulgaricus*, until a total acidity of 0·78—1·5% is developed, after which it is agitated and heated, first gradually to 52° and then rapidly to 85° in order to precipitate the casein and milk albumin in the form of non-adherent particles. The product is then evaporated *in vacuo* at a low temperature (60—65°), until it contains about 28% of solids, and its lactic acidity, which acts as a sterilising agent, is increased up to 6%. F. R. Ennos.

Manufacture of oleomargarine. H. Leroudier (E.P. 252,369, 18.5.26. Conv., 20.5.25).—The primary mass of melted oleomargarine fats and oils, either pure or emulsified with not more than 5% of water, is mixed with a suspension of fats or oils in a much larger quantity of water, the use of natural or artificial milk being excluded. F. R. Ennos.

Preservation of liquid eggs or egg constituents. United Yeast Co., Ltd., W. Salmon, and F. Jackson (E.P. 270,052, 18.3.26).—The liquid eggs, or their constituent yolk or albumin, are mixed below 21° with 1% ammonia solution in such amount, which is varied according to the length of time it is desired to preserve the eggs, that the free ammonia remaining when they are required for use shall be approximately 0·25%. The mixture is then transferred to air-tight containers which are hermetically sealed. F. R. Ennos.

Apparatus and continuous method for cooking cheese. C. Doering and H. H. Doering (E.P. 270,046, 8.3.26).—The cheese, which is cut into small particles in one chamber, is moved by means of a worm screw-conveyor to the second chamber, where it is cooked at about 60° by introduction of steam at various points, the worm-screw in this chamber being hollow to permit the circulation of steam therein. The mass, after mixing and removal of excess of moisture, is treated with flavouring and ageing ingredients and passed through a third or blending chamber maintained at 60°. Thence it is delivered in a continuous stream, from which solid particles are separated by reticular means, to an agitating vessel, in which it is aerated with oxygen or carbon dioxide under pressure. F. R. Ennos.

Vitamin preparation. M. Winckel, Assr. to H. G. Weicker (U.S.P. 1,624,154, 12.4.27. Appl., 11.8.25).—Skim milk is inoculated with a culture of sour milk bacillus *e.g.*, *B. Bulgaricus*, and allowed to ferment, with gradual addition of calcium carbonate to keep the acidity down, until the milk coagulates. Yeast is then added and fermentation continued for another 2—3 hrs. The product is afterwards treated with fruit juices, if desired, neutralised with more calcium carbonate, and dried at a low temperature. F. R. Ennos.

Preparation of non-fermented beverages. F. Hostettler (U.S.P. 1,625,542, 19.4.27. Appl., 23.8.26. Conv., 17.9.25).— Fresh fruit must is treated with sulphur dioxide under pressure, which cleanses it and renders it capable of being stored for an indefinite period without pasteurisation or refrigeration. When required for use it is desulphurated by exposure at a relatively low temperature to a current of humidified air in a partial vacuum. F. R. Ennos.

Artificial preparation of natural mineral waters. O. Warburg (E.P. 259,612, 9.10.26. Conv., 9.10.25).—

A solution in water is made of predetermined proportions of such substances, *e.g.*, sodium bicarbonate and sodium bisulphate, as will react to produce a content of carbon dioxide the same as that of the natural mineral water. Any components which are missing or present in insufficient quantities are subsequently added in the required proportions. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 18,700 of 1891.] F. R. Ennos.

Production of an edible protein product. J. W. M. Bunker, Assr. to American Protein Corp. (U.S.P. 1,624,036, 12.4.27. Appl., 19.6.25).—Blood serum, prepared from ox blood by removal of the red corpuscular matter and fibrin, is mixed with 15—20% of carbon tetrachloride at about 35° to remove the fatty substances, and allowed to settle. The carbon tetrachloride is separated and recovered by distillation, whilst the serum is dried to a granular powder by spraying into heated air. F. R. Ennos.

Manufacture of pectin products. D. R. Nanji and F. J. Paton (E.P. 269,952, 28.12.25).—Pectinous substances, *e.g.*, apple pomace, beet cossettes, etc., after removal, if necessary, of any excessive amount of natural acidity by electro-dialysis, are continuously extracted at 120° under pressure with a dilute (0·1—0·5%) solution of ammonium tartrate, sulphate, or citrate, or such substances capable of reacting with pectins by double decomposition to form a soluble salt of pectic acid with precipitation of an insoluble calcium salt. After clearing and filtration or centrifuging, the concentrated extract is treated with a small amount (0·2%) of calcium in the form of a soluble salt to augment its jellifying power. The process is applicable to the preparation of preserves from dried fruits in which a considerable part of the pectinous material has been rendered insoluble by drying. F. R. Ennos.

Preparation of baking and effervescent powders etc. I. G. Farbenind. A.-G. (G.P. 437,154, 10.1.25).—The tartaric acid or potassium bitartrate constituent of baking or effervescent powders etc. is partly or entirely replaced by fumaric acid or its acid salts. F. R. Ennos.

Production of coffee free from caffeine. F. Kündig (U.S.P. 1,629,512, 24.5.27. Appl., 1.5.25. Conv., 6.5.24).—See F.P. 597,392; B., 1926, 297.

Apparatus for recovering alcohol from vapours generated in bakers' ovens. M. Andrusiani (E.P. 270,358, 6.11.25).—See F.P. 603,218; B., 1926, 766.

Lævulose product (U.S.P. 1,616,170).—See XVII.

Extracting cell juices (F.P. 606,932).—See XVII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

[Analysis of] Rumanian industrial alcohol. A. Zaharia and D. Motzoc (Bul. Soc. Chim. România, 1926, **8**, 57—91).—A critical discussion of the methods which have been proposed for the analysis and purification of alcohol leads to the following procedure. Esters are removed and determined by saponification (Girard-Cuniasse method; cf. "Manuel pratique de l'Analyse des Alcools," 1899). The following improved Girard-Cuniasse method is the only one which removes acetaldehyde completely from alcohol. The alcohol is boiled under a reflux condenser with 4 g. of *m*-phenylenediamine hydrochloride and 4 g. of aniline hydrogen phosphate per 100 g. of alcohol. The mixture is then distilled under reduced pressure (15 cm.). Aldehyde is determined by colorimetric methods using Schiff's reagent; a solution of this reagent containing 1·5% of fuchsin is twice as sensitive as one containing 0·15%, and is quite stable. The total acidity is determined by direct titration with sodium hydroxide using phenolphthalein as indicator. Furfuraldehyde is determined by Jorissen's method (Ber., 1880, **13**, 2433) and higher alcohols by Komarovsky's method (B., 1903, 1259). The amount of impurities in Rumanian industrial ethyl alcohols is, up to a concentration of 91°, independent of the concentration of the alcohol, but, except in the case of esters, depends on the raw material used. Thus, alcohol extracted from molasses has a high acidity and aldehyde content; furfuraldehyde and higher alcohols occur in relatively large quantities in alcohol manufactured from cereals, but not from potatoes. The amount of impurities varies in parallel with the amount of higher alcohols. Most of the absolute alcohols examined contained furfuraldehyde and especially esters and acids. The impurities are independent of the nature of the raw material. Rectified alcohol derived from potatoes is the purest, and that from a mixture of maize and molasses the least pure. Industrial rectified alcohol is superior to that prepared in agricultural establishments. S. K. Tweedy.

Analysis of *iso*propyl alcohol. W. H. Simmons (Perf. Ess. Oil Rec., 1927, **18**, 168—169).—From an examination of various methods for the determination of acetone, the hydroxylamine method (cf. Bennett; B., 1922, 391 A) gives the most satisfactory results in the presence of *iso*propyl alcohol. The method of Bennett and Garratt (B., 1925, 264) for the determination of ethyl alcohol and Durrans' method (B., 1924, 731) for the detection of water are recommended. E. H. Sharples.

Standardisation and stabilisation of aconite preparations. III. E. E. Swanson and C. C. Hargreaves (J. Amer. Pharm. Assoc., 1927, **16**, 296—301).—Aconite tinctures and fluid extracts deteriorate rapidly within one year, and this loss in activity can be partially or totally prevented by the addition of sufficient acetic or hydrochloric acid to raise the p_H value to 2·5—3·0. For the standardisation of aconite preparations the biochemical method, in which the toxic dose for guinea-pigs or white mice is measured, is more reliable than the chemical method based on the ether-soluble alkaloid content (cf. Swanson and Walters, *ibid.*, 1923, **12**, 957; Swanson, *ibid.*, 1924, **13**, 1108). The guinea-pig and white mice methods agree well with standard aconite preparations, but not with deteriorated samples. W. J. Powell.

Alkaloidal content of British Columbian-grown *Hydrastis canadensis* and *Atropa belladonna*. R. H. Clark and (Miss) A. G. Winter (Trans. Roy. Soc., Canada, 1926, [iii], **20**, III, 307—312).—The air-dry roots of *Hydrastis canadensis* grown in the Botanical Gardens of the University of British Columbia contained

1·5% of hydrastine and 1·7% of berberine. *Atropa belladonna* grown in the same place contained in the leaves 0·60% and in the roots 0·67% of total alkaloids. R. CUTHILL.

Pharmaceutical incompatibility of camphor. D. MIGLIACCI and A. CALÒ (Annali Chim. Appl., 1927, **17**, 209—213).—The system camphor-ethylurethane shows a eutectic at 28·1°, corresponding with 49% of camphor. Above this temperature the mixtures are only pasty or liquid, whilst below it crystalline mixtures are formed. T. H. POPE.

Isomerisation of hydrocarbons by phenols. S. KONDAKOV (Parfumerie moderne, 1926, **19**, 212—223; from Chem. Zentr., 1927, I, 193—194).—The conversion of pinene (mixture of α- and β-pinenes from turpentine of various origins) into camphene by heating with picric acid, 2 : 4 : 5-trinitro-*m*-cresol, trinitrothymol, 2 : 4-dinitrophenol, 2 : 6-dinitro-*p*-cresol, dinitro-*o*-cresol, *p*-nitrophenol, 4-nitro-*m*-cresol, tribromophenol, methyl salicylate, resorcinol, *o*-cresol, phenol, etc., is investigated. When pinene (100 pts.) is heated with picric acid (20 pts.) a brisk reaction occurs at 145° (water being given off), and continues for some time without further application of heat. Bornyl picrate, m.p. about 130—138°, contaminated with a little diterpene, is precipitated, and a colourless oil is obtained on steam distillation. Not more than 10% of monocyclic terpenes (dipentene and terpinene) and 3% of diterpene are formed. Below 140° there is no isomerisation to camphene, part of the pinene remaining unchanged and the nopinene being converted into orthopinene. Bornyl picrate is hydrolysed by atmospheric moisture or by aqueous or alcoholic alkalis, and by treatment with organic bases (*e.g.*, pyridine) gives camphene. The first three fractions (b.p. 157—167°) from the steam distillate have a rather higher b.p. and lower rotatory power than the original pinene, give by oxidation with alkaline permanganate Wagner's camphenecamphoric acid, m.p. 135—136°, and no nopinic acid, and contain up to 75% of camphene. The fourth fraction (b.p. 167—170°) contains 50% of camphene and 50% of dipentene; the fifth (b.p. 170—178°) is almost pure dipentene, and the last contains nitrogenous compounds having a musk odour and some borneol. When pinene is shaken with mercuric acetate solution in the dark there is formed a pasty, pale yellow complex compound which becomes black in the light. Mixtures of pinene and camphene react with mercuric acetate more slowly, and the presence of 75% of camphene prevents reaction. Sobrerol and unchanged camphene result from the action of mercuric acetate on the hydrocarbon mixture obtained by a single treatment of pinene with picric acid. A second picric acid treatment gives almost exclusively low-boiling hydrocarbons, and very little dipentene, diterpene, or bornyl picrate. Picric acid reacts less vigorously with the nopinene fraction (b.p. 160—163°) of French and American turpentines; the products resemble generally those of the orthopinene fractions of the same oils, but differ in optical properties. Other tri- and di-nitrophenols give results similar to those with picric acid, but salts corresponding with bornyl picrate are not obtained with dinitrophenols. Mononitrophenols give a smaller yield of camphene, no bornyl ether, no steam bubbles, no monocyclic terpenes, and only traces of diterpenes. 2 : 4 : 5-Trinitro-*m*-tolyl bornyl ether is formed in greater quantity than the picrate, and is hydrolysed less rapidly; it yields camphene when heated. From halogenated phenols and pinene the yield of camphene is not more than 20%, and is independent of temperature (120—240°), time of heating (1—50 hrs.), and pressure (up to 4 atm.); no monocyclic terpenes, only traces of diterpenes, and no appreciable quantities of ester are formed. Phenol ethers isomerise at most 20—25% of pinene to camphene; below 200° no diterpene is produced, and at 240° under high pressure only traces of dipentene, terpinene, and diterpene. The increase in rotatory power indicates that the orthopinene content has increased, whilst the nopinene is probably isomerised to a mixture of camphene and *l*-α-pinene. The varying optical activity of pinenes from different sources is to be attributed not only to the proportions of *l*- and *d*-α-pinenes or *l*-α and -β-pinenes, but also to the proportion of orthopinene. The isomerisation of pinene to camphene by means of phenols and phenol derivatives probably occurs in nature, camphene and phenols being commonly found together.
C. HOLLINS.

Saccharin drying. E. BELANI (Chem.-Ztg. 1927, **51**, 261—262).—Describes the most recent improvements in rotary dryers for saccharin. L. M. CLARK.

Pinene content of Indian turpentine. H. M. MULANY and E. R. WATSON (J. Indian Chem. Soc., 1926, **3**, 258—259).—The results of various tests for pinene in the fraction (8·6—25%) of b.p. 155—160° of Indian turpentine oil, compared with those obtained with samples from other sources, show that, contrary to the statement of Simonsen (B., 1920, 581 A), the pinene content of this oil is very small. It contains a small quantity of Δ^3-carene, detected as the nitrosate.
J. W. BAKER.

Function and formation of essence of mint by the secretive hairs of the plant. J. RIPERT (Chim. et Ind., 1927, **17**, 203—208).—From investigations on the formation of essence of mint during the growth of the plant (*Mentha piperita*) under various conditions, it is concluded that the essence is a product of de-assimilation, its formation being dependent on the balance existing between the forces of decay and growth. A. J. HALL.

Essential oil content of some British Columbian-grown mints. R. H. CLARK (Trans. Roy. Soc. Canada, 1926, [iii], **20**, III, 469—473).—Various species of mint have been grown in the Botanical Gardens of the University of British Columbia. *Mentha piperita* and *M. viridis* yielded a percentage of oil which was approximately normal, but the yield per acre was very poor, largely due to a rust. *M. pulegium*, *Tanacetum vulgaris*, and *Monarda fistulosa*, mollis, grew well and gave good yields of oil. *Thymus vulgaris* grew well, but gave a rather low yield of oil. R. CUTHILL.

Essential oils from Greece. J. GASOPOULOS (Arch. Pharm., 1927, **265**, 41—44).—The following data are for oils from cultivated plants. From *Rosa damascena*, 1922 and 1923 crops: d_{15}^{30} 0·8724, d_{15}^{25} 0·8667; α_D —2°, —3°; n^{29} 1·4660, n^{30} 1·4640; setting-point, 16°, 19°; acid value, 1·4, 1·0; saponif. value, 16·5, 16·2; saponif. value, after acetylation, 255·9, 243·6; alcohol,

$C_{10}H_{18}O$, 87·4%, 81·9%; ester, $C_{10}H_{17}\cdot CO_2Me$, 5·7%, 5·67%. From *Laurus nobilis*, L. (three samples): d^{15} 0·9200, 0·9179, 0·9198; α_D —17·2°, —16·8°, —18·2°; n_D 1·4669, 1·4600, 1·4670; acid value, 1·5, 1·4, 1·4; saponif. value, 34·5, 31·4, 29·4; ester value, 33, 30, 28. From *Lavandula vera* (three samples): d 0·889, 0·881, 0·884; α_D —7°, —4°, —8°; n_D 1·4621, 1·460, 1·461; ester value, 86, 83, 100; linalyl acetate, 30%, 29%, 35% (these values are low because distillation was conducted by direct heating). From *Rosmarinus officinalis* (three samples): d 0·991, 0·908, 0·912; α_D + 1·51°, + 1·00°, + 1·40°; acid value, 0·3, 0·5, 0·3; ester value, 3, 6, 4; bornyl acetate, 1·05%, 2·10%, 1·4%; ester value after acetylation, 24, 20, 18; saponif. value, 3·3, 6·5, 4·3; borneol, 6·8%, 5·6%, 5·0%.

W. A. SILVESTER.

Essential oil content of *Chamaecyparis nootkatensis*. R. H. CLARK and C. C. LUCAS (Trans. Roy. Soc. Canada, 1926, [iii], **20**, III, 423—428).—The leaves of *Chamaecyparis nootkatensis* when distilled in steam yield 0·7—2·0% of their air-dry weight of a dextrorotatory oil with d^{20} 0·855—0·8880, b.p. 155—280°, n_D^{20} 1·472—1·474, and saponif. value 10—30. Limonene, α-pinene, *p*-cymene, and probably β-pinene and sabinene are present in the oil.

R. CUTHILL.

Phytopharmacological study of digitalis assay. D. I. MACHT and J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1927, **16**, 210—218).—Methods of biological assay of digitalis preparations employing animals are unsatisfactory owing to variations in the susceptibility of individual animals etc. It is now proposed to employ seedlings of *Lupinus albus*, the inhibition of growth of the roots in a nutrient solution for plants (Shive solution) having been found to be proportional to the concentration of the digitalis solution. The phytotoxic curve obtained has been calibrated in terms of "cat units" by careful comparison with the cat method, and a method for the examination of digitalis tinctures is described, an allowance being made for the toxic effect of the alcohol present in such tinctures by introducing equivalent amounts of alcohol into the control experiments.

C. O. HARVEY.

Determination of cineole in essential oils. I. Cajuput and eucalyptus oils. REP. ESSENTIAL OIL SUB-COMM. TO THE STANDING COMM. ON UNIFORMITY OF ANALYTICAL METHODS (Analyst, 1927, **52**, 276—279).—The British Pharmacopœia method is regarded as unsatisfactory, and the *o*-cresol f.p. method is recommended. 3 g. of the oil and 2·1 g. of melted *o*-cresol are weighed successively into a test tube to an accuracy of one small drop (about 0·02 g.), a thermometer graduated in fifths of a degree is inserted, the mixture stirred, and the highest reading taken. The tube is then warmed and inserted into a wide-mouthed bottle through a cork and allowed to cool slowly until the previous reading is reached or crystallisation begins, when stirring and rubbing is begun and continued until there is no further rise of temperature, the highest point reached being taken as the f.p. The test should be repeated until two concordant results are obtained. If less than 50% of cineole is present pure cineole or a high-content oil may be added, or, if the mixed liquids do not crystallise in the first attempt, 5·1 g. of the pure recrystallised additive product, *o*-cresol–cineole, may then be added. A table of mean f.p. values for percentages of cineole from 45·6 to 100 is given.

D. G. HEWER.

Valuation of valerian root and valerian extract. G. J. ÖSTLING (Dansk. Tidsskr. Farm., 1927, I., 363—374).—The nature of the active therapeutic principle in valerian root is uncertain, the quality of the material being usually judged on the appearance and odour of the drug and of the extract prepared from it. By boiling the drug with a mixture of alcohol and water (2 : 1) for ½ hr., filtering, evaporating, and determining the weight of the dry residue, results were obtained which agreed with those obtained from extracts made in the usual way by percolation. The method of Katz is described in which 25 c.c. of the extract are evaporated to dryness with 1 c.c. of 33% sodium hydroxide solution. The dry residue is dissolved in 100 c.c. of water, 20 c.c. of 25% phosphoric acid are added, and the mixture is distilled. 110 c.c. of distillate are collected and titrated with 0·1*N*-alkali, using phenolphthalein as indicator, the acid found being reckoned as valeric acid. Various specimens of the drug from different countries all give approximately equal acid values when thus examined, despite the fact that Japanese valerian contains 6% of ethereal oil as against 1% for the European drug. Injection experiments on frogs were carried out, using alcoholic valerian extracts, each extract prior to employment being evaporated to ensure removal of the alcohol, in order to eliminate errors due to the physiological action of this substance. The results showed that extracts prepared according to the method of the Finnish Pharmacopœia using alcohol (2 pts.) and water (1 pt.) were more effective physiologically than those prepared by percolation with a mixture of water (3 pts.) and alcohol (1 pt.). Valerian extracts should be prepared by the former method, despite the fact that an extract of this type becomes turbid on the addition of water, whilst those prepared with weaker alcohol remain clear. Further experiments showed that extracts prepared from dried valerian root were apparently quite as effective physiologically as those made from fresh-cut valerian, although the results were not absolutely conclusive.

H. F. HARWOOD.

Crismer test for essential oils. A. ANGELETTI (Annali Chim. Appl., 1927, **17**, 263—268).—The critical temperature of solution in alcohol, suggested by Crismer (B., 1896, 300; 1897, 70; 1904, 451) as a distinctive physical constant, is applied to essential oils. The results are given for mixtures of each of a number of the oils with equal volumes of alcohol (d 0·8882 or 0·8899 at 15°) (cf. Asbôth; B., 1897, 640) and for mixtures in various proportions with alcohol of d 0·8882; the critical solution temperature rises with the percentage of the essential oil in the mixture. The test furnishes useful indications of the degree of purity of the oils.

T. H. POPE.

Dehydration of alcohol. PETRLIK.—See II.

Alcohol from cellulose etc. MUTTI and MONTALTI.—See V.

PATENTS.

Preparation of aromatic carboxylic esters of alkoxy- and dialkoxy-aryldialkylaminopropyl alcohols. C. MANNICH (G.P. 437,925, 23.4.24).—The alcohols are treated with benzoyl or nitrobenzoyl chloride, giving basic esters which have powerful local anæsthetic action and low toxicity. β-*Dimethylamino*-β-3 : 4-*methylenedioxyphenyl*iso*propyl alcohol*, m.p. 67—68°, prepared from *iso*safrolebromohydrin and dimethylamine, when heated with benzoyl chloride for 1 hr. at 125°, yields the O-*benzoate*, m.p. 81—82° (*hydrochloride*, m.p. 206—208°). The O-p-*nitrobenzoate* (*hydrochloride*, m.p. 216°), similarly obtained, is reduced by tin and hydrochloric acid to the p-*aminobenzoate*, m.p. 68°. β-*Diethylamino*-β-3 : 4-*methylenedioxyphenyl*iso*propyl alcohol*, b.p. 170—175°/14 mm., gives an O-*benzoate* (*hydrochloride*, m.p. 205—207°), and an O-p-*nitrobenzoate* (*hydrochloride*, m.p. 220°). From β-*piperidino*-β-3 : 4-*methylenedioxyphenyl*iso*propyl alcohol*, m.p. 77°, an O-*benzoate* (*hydrochloride*, m.p. 204°), an O-p-*nitrobenzoate* (*hydrochloride*, m.p. 208°), and an O-p-*aminobenzoate*, m.p. 70°, are obtained. β-*Dimethylamino*-β-*anisyl*iso*propyl alcohol*, b.p. 162°/17 mm., from anetholebromohydrin, gives an O-p-*nitrobenzoate* (*hydrochloride*, m.p. 205°) and an O-p-*aminobenzoate*, m.p. 159°. The O-p-*nitrobenzoate* (*hydrochloride*, m.p. 198°) of β-*dimethylamino*-β-3 : 4-*dimethoxyphenyl*iso*propyl alcohol* (from *iso*eugenolbromohydrin) is described. C. HOLLINS.

Iodising pyridine derivatives. S. G. S. DICKER. From DEUTSCHE GOLD- & SILBER-SCHEIDEANSTALT, VORM. ROESSLER (E.P. 246,501, 22.1.26).—Pyridine derivatives containing an amino- or hydroxy-group in position 2 or 4 are treated with iodine in the presence of a mild alkali (potassium carbonate, borax, etc.), caustic alkalis being avoided. The preparation of iodo-2-aminopyridine, 3-*iodo*-5-*nitro*-2-*hydroxypyridine*, m.p. 203°, and 5-iodo-2-hydroxypyridine, m.p. 275°, is described. The compounds have therapeutic value. C. HOLLINS.

Production of 5-iodo-2-aminopyridine. C. RÄTH (E.P. 246,842, 26.1.26. Cf. E.P. 251,578; B., 1926, 608).—The method of Magidson and Menschikov (A., 1925, i, 301) is improved by the use of 10—30% alkali in place of the 3% alkali used in purifying the product of iodination of 2-aminopyridine with iodine in sodium or potassium iodide solution, and the pure 5-iodo-2-aminopyridine is crystallised from water instead of being extracted with ether. C. HOLLINS.

Manufacture of asymmetrically acylated amino-derivatives of arylarseno-compounds. G. NEWBERY, and MAY & BAKER, LTD. (E.P. 269,647, 19.1.26).—The reaction $Ar\cdot As_2\cdot Ar + Ar'\cdot As_2\cdot Ar' \rightarrow 2Ar\cdot As_2\cdot Ar'$, where Ar is a diacyldiaminoaryl and Ar′ a diaminoaryl group, either or both of which may contain other substituents, occurs when the two substances are dissolved in a common solvent and a precipitant for the product is added. By adding ether or excess of methyl-alcoholic hydrogen chloride to a mixture of 3 : 3′-diamino-5 : 5′-diacetamido-4 : 4′-dihydroxyarsenobenzene (prepared by reduction of nitrated 3-acetamido-4-hydroxybenzenearsinic acid with hyposulphite) and 3 : 3′-diamino-4 : 4′-dihydroxyarsenobenzene dissolved in methyl alcohol with the aid of a little methyl-alcoholic hydrogen chloride, there is precipitated the dihydrochloride of 3 : 3′-diamino-5-acetamido-4 : 4′-dihydroxyarsenobenzene. 3-Amino-3′-acetamido-4 : 4′-dihydroxyarsenobenzene is prepared by adding acetic acid to a solution of 3 : 3′-diamino-4 : 4′-dihydroxyarsenobenzene and its diacetyl derivative in dilute sodium hydroxide, and 3-amino-3′ : 5-diacetamido-4 : 4′-dihydroxyarsenobenzene is similarly obtained from 3 : 3′-diamino-5 : 5′-diacetamido-4 : 4′-dihydroxyarsenobenzene and 3 : 3′-diacetamido-4 : 4′-dihydroxyarsenobenzene. The reduction of 3-nitro-5-acetamido-4-hydroxybenzenearsinic acid with sodium hyposulphite and magnesium chloride to the diaminodiacetamidodihydroxyarsenobenzene is described. From 3 : 3′ : 5 : 5′-tetra-acetamido-4 : 4′-dihydroxyarsenobenzene, prepared by reduction of 3 : 5-diacetamido-4-hydroxybenzenearsinic acid, and 3 : 3′-diamino-4 : 4′-dihydroxyarsenobenzene there is obtained 3-amino-3′ : 5′-diacetamido-4 : 4′-dihydroxyarsenobenzene. C. HOLLINS.

Manufacture of asymmetrical arylarseno-compounds. G. NEWBERY, F. J. PAXON, and MAY & BAKER, LTD. (E.P. 270,091, 7.5.26).—The processes of E.P. 11,709 and 11,901 of 1911 (B., 1912, 256), namely reduction of mixtures of two different arylarsinic acids etc., or condensation of arylarsines with a differently constituted arylarsenious oxide or dichloride, are applied to the manufacture of unsymmetrical arsenobenzenes containing in one of the nuclei a glycinamide group. Sodium phenylglycinamide-4-arsinate, $NH_2\cdot CO\cdot CH_2\cdot NH\cdot C_6H_4\cdot AsO(ONa)_2$, is reduced to the arylarsenious oxide, which is converted into the dichloride hydrochloride and allowed to react in aqueous solution with 3-amino-4-hydroxyphenylarsine hydrochloride to give 3-*amino*-4-*hydroxyarsenobenzene*-4′-*glycinamide*. The same product results when a mixture of phenylglycinamide-4-arsinic acid and 3-amino-4-hydroxybenzenearsinic acid is reduced with sodium hyposulphite and magnesium chloride. 3-*Acetamido*-4-*hydroxyarsenobenzene*-4′-*glycinamide*, 3-*amino*-5-*acetamido*-4-*hydroxyarsenobenzene*-4′-*glycinamide*, and 5-*acetamido*-2-*hydroxyarsenobenzene*-4′-*glycinamide* are similarly obtained by reduction of appropriate mixtures of arylarsinic acids. The products have therapeutic value, and in the form of monosodium salts are suitable for injection. C. HOLLINS.

Preparation of artificial medicinal waters [from coal]. H. W. A. BRANCO (E.P. 269,660, 4.5.25. Cf. G.P. 432,472; B., 1927, 265).—The ashes from lignites, coal, or peat are extracted with water; if desired the extracts may be further treated by the addition of salts (such as carbonates) or gases (such as carbon dioxide) or both. R. A. A. TAYLOR.

Manufacture of carbamide from cyanamide. J. BRESLAUER and C. GOUDET, Assrs. to SOC. D'ÉTUDES CHIM. POUR L'IND. (U.S.P. 1,630,050, 24.5.27. Appl., 27.1.23. Conv., 4.2.22).—See E.P. 192,703; B., 1923, 1152A.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Oxidising agents in the study of the sensitivity of photographic emulsions. W. CLARK (Brit. J. Phot., 1927, 74, 227—228, 243—245).—Preliminary

experiments are described, based on the following considerations: it is assumed that the sensitive nuclei in the grains consist of silver sulphide, and the latent image of silver sulphide plus silver. If, then, an oxidiser can be found which will attack silver and not silver sulphide, it should not appreciably desensitise an ordinary unexposed plate, but should desensitise a pre-exposed plate. On the other hand, with an oxidiser which will attack both silver and silver sulphide, desensitisation should occur both with and without preliminary exposure of the plate, but perhaps more readily in the case of pre-exposure since silver would be more readily attacked than silver sulphide. A solution containing potassium chromate, chrome alum, and sulphuric acid was used as the oxidiser of high potential. The desensitising properties of solutions of potassium persulphate, potassium permanganate, chrome alum, copper sulphate, potassium ferricyanide, uranium nitrate, sulphuric acid, and ammonium molybdate were also investigated. Potassium persulphate was chosen as the oxidiser of low potential for testing the above views. It was found that desensitisation is more pronounced the higher the oxidation potential of the desensitiser used. Chromate–chromic–sulphuric acid mixture desensitises very markedly, and the lowest speed attained was much lower than in the earlier experiments using plain chromic acid solution (B., 1923, 522 A, 630 A). Pre-exposure was practically without effect, and the solution attacks both colloidal silver sulphide and colloidal silver, the latter much more readily. Acidified persulphate desensitises, but less markedly than do the solutions of chromium salts, and with pre-exposure an increased desensitisation occurs, depending on the time of treatment. The acid persulphate attacks colloidal silver, but is without action on colloidal silver sulphide. Since persulphate, which does not attack silver sulphide, desensitises an unexposed plate, it is concluded that sensitivity cannot be due to silver sulphide alone, but that there is present before exposure something which is attackable by persulphate. The results can be explained on the assumption that the sensitive centres contain both silver sulphide and silver. The older and newer theories of the nature of sensitivity can thus be reconciled. Persulphate exerts a marked destructive effect on the latent image, the characteristic curves after persulphate treatment between exposure and development being similar in form to those obtained in ordinary persulphate reduction of the developed image. With desensitisation by chromate–chromic–sulphuric acid mixture there is a progressive decrease in gamma with increasing time of treatment, a result which is the reverse of that found in the case of plain chromic acid solution. The decreasing gamma is shown not to be due to retardation of the development rate as a result of tanning of the gelatin by the chromium salts. Hardening with chrome alum increases the rate of diffusion of certain substances in gelatin gels; formaldehyde in corresponding cases causes a retardation of the rate of diffusion. The effects on the speed of normal and desensitised plates of bathing in solutions of bicarbonate, acetic acid, and sodium bisulphite, and in alcohol are also recorded. All the results refer to the Ilford "Special Rapid" plate. W. CLARK.

Silver sulphide sensitising by bathing. S. E. SHEPPARD and E. P. WIGHTMAN. (Phot. J., 1927, **67**, 219—226).—High- and low-speed commercial plates showed no increase in speed on treatment in allylthiocarbamide solution followed by alkali, and fog could not readily be avoided. With a coarse-grained, low-speed, multilayer plate prepared from gelatin from which the sensitisers had been largely removed, an increase of speed could be obtained, and a change in colour sensitivity was observed. To avoid the difficulties met in the case of multilayer plates owing to the time taken for diffusion from the top layer of the emulsion to the bottom layer, thinly coated plates were studied. With increasing concentration of sensitiser the sensitivity increases to a maximum, and there is possibly an appreciable extension of the spectral sensitivity, an observation which is not in entire agreement with previous results, and needs confirmation. With variation of the hydrogen-ion concentration of the allylthiocarbamide sensitising bath in a two-stage procedure of sensitising (*i.e.*, allylthiocarbamide bath followed by an alkali bath) from p_H 2 to p_H 4, the degree of sensitising was about equal. An apparent increase of speed at p_H 5 may be due to fog. Soluble bromide decreases the amount of sensitising. Sensitisation of chromic acid desensitised plates and desensitisation of sensitised plates were also studied. Sensitising after desensitising produces a marked amount of fog. The existence of a maximum in sensitising is ascribed to an ionic lattice strain and ionic deformation, together with a competition among the sensitive specks for the silver produced photochemically. The experiments do not allow of a decision between the "concentration speck" theory and the halogen acceptor theory of Hickman (B., 1927, 126). W. CLARK.

Sensitivity of silver halide grains in relation to size. S. E. SHEPPARD and A. P. H. TRIVELLI (Phot. J., 1927, 67, 284—287, and J. Franklin Inst., 1927, **203**, 829—833).—Renwick's finding (B., 1924, 770) that in iodobromide emulsions investigated the larger grains in any one emulsion had a higher content of silver iodide than the smaller ones, is confirmed with other high-speed iodobromide emulsions. In a pure bromide emulsion it was found that the larger grains are relatively more sensitive than the smaller. The greater proportion of iodide is, thus, not a necessary condition for the relative greater sensitivity of the large grains in an iodobromide emulsion. W. CLARK.

Sensitising specks on silver halide grains. S. E. SHEPPARD, A. P. H. TRIVELLI, and E. P. WIGHTMAN (Phot. J., 1927, **67**, 281—283).—By bathing a large-grained pure bromide emulsion in allylthiocarbamide followed by sodium carbonate, it was found that specks of silver sulphide, visible in the microscope, were formed at points on the grain surfaces. Carbonate alone, allylthiocarbamide alone, and the filtered light used in photomicrographing did not give such specks. The specks obtained were quite distinct in manner of formation and appearance from those known to be formed by the action of white light. The observations give direct evidence that the silver sulphide formed on

grains during emulsion making exists as specks and not as a uniform distribution over the grain surfaces.
W. Clark.

Photographic reversion. V. P. Lubovich (J. Opt. Soc. Amer., 1927, **14**, 309—311).—Examples of photographic reversion due to over-exposure are given. For each kind of emulsion at a given wave-length and intensity there is a definite time of exposure for maximum density of developed image. If this is exceeded a diminished density results, and with longer exposures the image may be completely destroyed and thus appear reversed against the general background. Still longer exposure may produce a second maximum, but of less density than the first. It is suggested that this repeated reversion is analogous in character to a damped vibration.
C. J. Smithells.

Relation between time and intensity in photographic exposure. V. L. A. Jones, V. C. Hall, and R. M. Briggs (J. Opt. Soc. Amer., 1927, **14**, 223—232; cf. B., 1926, 566, 1030).—New data corroborate the previous views on effect of time of development on the failure of the reciprocity law and on the shift of optimal intensity. From the study of a large number of emulsions it is concluded that there is no silver bromide or bromoiodide emulsion for which Kron's "curves of constant density" in the region of optical intensity may not be represented by the Kron–Halm equation (Monthly Notes Roy. Astr. Soc., 1915, **75**, 150). Different developing agents appear to have no influence on the extent of the reciprocity failure, but more data on this point are required.
L. F. Gilbert.

Daylight developers. K. Binder (Phot. Korr., 1927, **63**, 111—112).—The salts of ferritripyrocatechol-oxides (A., 1912, i, 184) are deep red in colour, and can be used as developers in daylight. Addition of alkali is necessary, and sulphite should also be present. After plates have been in the developer for some time, they can be taken out for visual examination in daylight. Developed and fixed plates are quite colourless and free from fog.
W. Clark.

Gaslight paper and sulphur toning. P. Wiegelb (Phot. Ind., 1927, 470—472).—Toning of gaslight papers with sodium sulphide, barium sulphide, liver of sulphur, and thiocarbamide, after bleaching with ferricyanide–bromide, are described in detail. Variations by which a varying range of tones can be obtained are considered.
W. Clark.

Ferguson density comparator. W. B. Ferguson (Phot. J., 1927, **67**, 278—280).—A simple form of visual density meter is described, in which the densities are measured by comparison with a calibrated neutral wedge.
W. Clark.

Test for emulsion gelatin. R. Luther (Phot. Ind., 1927, 494—495).—Equal volumes of a test solution containing 20—25 g. of sodium hydroxide, 3—4 g. of lead nitrate, and 100 c.c. of water, and of 15—20% gelatin sol, are mixed and the mixture is heated in boiling water. A colourless flocculent precipitate first forms, and goes dark more or less quickly according to the amount of sulphur present in the gelatin. By comparing the velocity of formation and the amount of colour with that of standard gelatins of known photographic properties, conclusions can be drawn as to the photographic qualities of the gelatin.
W. Clark.

Patents.

***X*-Ray photographs and means for obtaining them.** L. Danin (G.P. 437,507, 16.10.25).—*X*-ray photographs obtained in the usual way may be coloured so as to show the relative densities of the objects photographed by soaking the plates, either before exposure or after exposing, developing, and fixing, in solutions, preferably alcoholic, containing substances sensitive to light together with different dyes as sensitisers.
A. R. Powell.

Photographic process. H. Wade. From Wadsworth Watch Case Co. (E.P. 269,806, 4.2.26. Cf. U.S.P. 1,587,269—1,587,274; B., 1926, 773).—Synthetic resinous products derived from benzaldehyde or formaldehyde, for example, by reaction with β-naphthylamine, are employed as media for forming an image. A sensitiser, such as iodoform or other halogen source, may be added.
W. Clark.

Photographic desensitiser. I. G. Farbenind. A.-G., Assees. of B. Homolka (G.P. 436,161, 14.12.24).—The green basic dyes, probably of the quinoneimine type, obtained by condensing 2-aminophenanthraquinone with *o*-aminodiphenylamines, are used as desensitisers. The *o*-aminodiphenylamine may be suitably substituted.
C. Hollins.

XXII.—EXPLOSIVES; MATCHES.

Patent.

Safety explosives. Soc. Gén. pour la Fabr. de la Dynamite (F.P. 613,089, 9.7.25).—The particles of ammonium nitrate or other constituents of the explosives are coated with a layer consisting of 25% of paraffin, 50% of vaseline, and 25% of resin.
L. A. Coles.

XXIII.—SANITATION; WATER PURIFICATION.

Effect of pipes of different metals upon the quality of water supplies. H. W. Clark (J. New England Water Works Assoc., 1927, **41**, 31—44).—Theories of corrosion are discussed and reference is made to early work of the author with regard to the solvent action of waters upon lead. Previous work showed that whilst in laboratory experiments oxygen was the chief factor in dissolving lead, in the conditions prevailing in service-pipes a dangerous amount of lead was not generally found in solution except when the supply contained a generous amount of carbonic acid, and was also soft. This is confirmed by more recent work in which it was noted that some ground-waters, containing dissolved oxygen to the extent of about 50% saturation and also quantities of carbonic acid, took up more lead than certain soft surface-waters nearly saturated with dissolved oxygen, but of relatively low carbonic acid content. Soft surface-waters, when new and long lengths of lead pipe are installed, may, however, for a time, before the formation of a suitable coating within the pipe, take up

dangerous amounts of lead into solution. Very little tin is taken from tin-lined pipes by any water. A greater amount of metal is taken into solution from iron pipes than from pipes of any other metal, and the greater the amount of oxygen present the greater the amount taken. An increase in the carbonic acid content in a surface supply by alum treatment increased the corrosion. Much zinc is taken into solution from galvanised iron pipes. Ground-waters have more corrosive action on zinc than surface-waters. Brass pipes yield much zinc, but generally only minute amounts of copper. Copper pipes yield about the same amount of copper as brass pipes. Less copper is taken up by hot water than by cold water. In experiments with new brass and copper pipes 25 ft. long, using four different waters and allowing periods of "standing" of from 8 hrs. to several months, nearly all the analyses showed copper to be present in quantities less than 0·10 pt. in 100,000. Little, if any, more copper was taken into solution by the ground- than by the surface-waters. Running samples from a new copper pipe 200 ft. long showed copper of an average amount of 0·0111 pt. in 100,000. Lead in average amounts of 0·04 pt. in 100,000 in drinking water will cause lead poisoning of certain individuals if habitually used; no instances of intestinal or other troubles arising from zinc in water have been recorded in the Massachusetts State. The p_H determination of a water has some significance in giving information of corrosive properties. W. T. Lockett.

Electrolytic chlorination at Sacramento filtration plant. H. N. Jenks (J. Amer. Water Works Assoc., 1927, **17**, 514—537).—A description of the electrolytic plant is given, together with details of operation and cost. Experience covering 2½ years proves that the manufacture of electrolytic chlorine in conjunction with the operation of a water-filtration plant, sewage disposal works, or industrial establishment is thoroughly feasible, and may often be of decided economic advantage in effecting a substantial saving in the cost of chlorination. The installation described consists essentially of six 600-amp. cells capable of producing 228 lb. of chlorine per 24 hrs. The average quantity of salt used is 4·60 lb. per lb. of chlorine produced, and the average amount of current required works out at 2·66 kw.-hrs. per lb. of chlorine. By the adoption of electrolytic chlorination a saving of 57% has been effected, due partly to the low unit cost of current, but more particularly to the high purchase price of liquid chlorine. W. T. Lockett.

Use of lime in water softening and purification. C. P. Hoover (Ind. Eng. Chem., 1927, **19**, 567—570).—Lime softening frees water from bacteria (especially if the lime is slightly in excess), turbidity, iron, colour, organic matter, and objectionable gases, and lessens the cost of filtration. The limitations to its use have been due to the incrustation of pipes etc. with carbonate owing to supersaturation with normal carbonates of calcium and magnesium, and to the formation of soluble complex basic carbonates of magnesium, which cause the alkalinity to be high. Recarbonation with carbon dioxide prevents such incrustation, whilst the soluble basic carbonates are dealt with in several ways, *e.g.*, by the hot process, since calcium and magnesium carbonates are less soluble at higher temperatures, or by the use of lime in excess (2—3 grains per gallon), to precipitate the magnesium almost completely, the excess sodium hydroxide being eliminated either by dilution with raw water or by carbonation and filtration. The addition of aluminium compounds is believed to convert soluble magnesium salts into insoluble magnesium aluminates, making it possible to remove them by settlement or filtration. Zeolites may be substituted for soda ash to remove permanent hardness after lime treatment, the water being carbonated to prevent deposition on the zeolite. The presence of sodium bicarbonate in water softened by zeolite makes it possible to mix with it water containing permanent hardness and not get an after-precipitation. W. G. Carey.

Lime in the treatment of pea cannery wastes. L. F. Warrick (Ind. Eng. Chem., 1927, **19**, 577—583).—The effluents from pea-canning factories become objectionable in odour in hot weather and also cause stream pollution. The oxygen demand of such waste can be reduced about 75% by screening followed by tank treatment with 7·25 lb. of lime and 3·25 lb. of ferrous sulphate per 1000 gals., but the chemically precipitated organic matter should be removed speedily, since a portion goes into solution when allowed to accumulate in the tank; the reduction in oxygen demand averaged only 34% when the organic matter was not removed. Sludge is pumped from the tank by a diaphragm pump and is dried on sludge beds, yielding a fertiliser containing 3·3% of available phosphoric acid. Aeration also serves still further to reduce the oxygen demand of the tank effluent. W. G. Carey.

Patents.

Settling tank. G. P. Kropp (E.P. 260,279, 21.10.26. Conv., 21.10.25).—The settling tank comprises, in combination, a chamber, supplied from several sides with separate streams of the liquid to be purified for the purpose of causing them to impinge against one another and provided with an outlet at the bottom, also a second chamber of tapering form, arranged with its converging bottom below that of the first one to receive the liquid on its discharge from the first chamber, and furnished with a sludge outlet and a surrounding overflow device to which the liquid rises after its purification. W. T. Lockett.

Treatment of dye effluents. Darco Sales Corp., Assees. of P. Mahler (E.P. 262,382, 18.6.26. Conv., 3.12.25).—Dye effluents are decolorised by adding activated carbon to the heated effluent, reagents being added, if necessary, to produce a flocculent precipitate of an inorganic hydroxide or to convert colloidal dyes into the molecular state. After settlement and decantation the carbon is recovered from the precipitate by an oxidising agent, such as a chlorate and hydrochloric acid, which also dissolves the precipitate. W. G. Carey.

Tanks and apparatus for aerating sewage and other foul liquids. R. Ames (E.P. 270,012, 4.2.26).

Bactericidal agents (G.P. 438,241).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 22, 1927.

I.—GENERAL; PLANT; MACHINERY.

Recovery of volatile solvents by "chemical washing." H. Wiesenthal (Chem.-Ztg., 1927, **51**, 373).—The "Cheminova" apparatus as used for the recovery of alcohol and ether vapours in the collodion artificial silk process is described. The air containing 20 g./cub. m. of vapour is passed through a dust filter to a series of towers, where it is treated with a suitable reagent. From this alcohol, ether, and water are recovered by distillation, and the distillate is fractionated.
C. Irwin.

Determination of pressure of liquefied gases within containers. R. Nitzschmann (Continental Met. Chem. Eng., 1927, **2**, 79).—Equations and charts defining the pressures developed between -10 and $+45°$ in containers of liquid chlorine in relation to the volume and the filling temperature and pressure indicate that, under certain conditions, the containers are advantageously filled at relatively high temperatures.
Chemical Abstracts.

Further applications of a mobilometer. H. A. Gardner and A. W. van Heuckeroth (Ind. Eng. Chem., 1927, **19**, 724—726).—A mobilometer is described in which the time taken to push a metallic disc (perforated or otherwise), suitably loaded with a known weight, through a given column of material is measured. Applications in the paint and varnish and in other industries are described. The time of "flow" usually varies appreciably when diluents are added to the material being tested.
S. K. Tweedy.

Patents.

Ball mill. P. L. Crowe (U.S.P. 1,627,487—8, 3.5.27. Appl., [a, b], 30.9.25).—A cylinder rotating on a horizontal axis is divided by a vertical screen into a grinding chamber containing steel balls and a blowing chamber containing fan blades. These latter induce a current of hot air, which carries pulverised coal from the grinding chamber through the screen into the furnace. In a modification of the apparatus the grinding chamber surrounds the blowing chamber.
T. S. Wheeler.

Grinding mill. J. R. Torrance (E.P. 270,465, 25.3.26).—In a roller mill for, *e.g.*, paint, the material is fed to one end of a pair of rolls and is not removed until it arrives at the other end, guiding and scraping blades being provided. The rolls are given a considerable differential speed, the roll with the higher speed being of larger diameter than the other. They may be either cylindrical or frusto-conical with equal and opposite tapers.
B. M. Venables.

Rotary drying apparatus. H. A. Marston (E.P. 270,503, 3.6.26).—Hot gases from a furnace pass first over the outside of a rotary drying drum and then through the interior, counter-current to the material. The drum is provided with one or more enlarged zones containing baffles and helical blades, which delay and mix the material and give the hot gases a whirling motion.
B. M. Venables.

Mixing and agitating machines and appliances. B. Goldman (E.P. 267,480, 8.2.27. Conv., 9.3.26).—In a tank containing liquid, which is set in motion by any desired means, baffles are suspended by cords or chains in such a way that the drag of the liquid will cause them to move upwards towards the surface of the liquid.
B. M. Venables.

Method and apparatus for leaching. A. B. Kennedy (U.S.P. 1,628,787, 17.5.27. Appl., 21.2.22).—A tank for leaching tannins etc. from bark has the bottom formed of a series of troughs of semicircular cross-section placed edge-to-edge. A paddle wheel rotating in each trough passes the material under treatment into the adjacent trough against the flow of extracting liquid, and prevents the material from rising to the surface of the liquid.
T. S. Wheeler.

Apparatus for dissolving and filtering minerals and other inorganic material. Comp. de Prod. Chim. et Électrométallurgiques Alais, Froges, et Camargue (F.P. 614,862, 21.4.26. Conv., 22.4.25).—A cylindrical vessel is divided into two sections by a filtering plate upon which the material is treated with the solvent. Means are provided for heating and agitating the material upon the plate.
L. A. Coles.

Filtration. E. A. Alliott, A. E. Hatfield, and Achille Serre, Ltd. (E.P. 270,461, 20.3.26).—To each batch of the soiled liquid flowing from a clothes-washing or cleaning machine a definite volume of filter-aid pulp is added at a constant rate. When the measuring vessel delivering to the pulp has emptied itself to a certain level, the vessel is refilled and the pulp diluted with fresh or soiled washing liquid, so that, the outflow remaining constant, the amount of filter aid added per sec. decreases in the later stages; this compensates for the fact that the washing liquid carries most dirt in the early stages. The dilution of the pulp may be effected several times. The apparatus comprises mainly a measuring vessel with internal float which governs the dilution, and another float chamber which prevents any vacuum in the pipe conveying the washing liquor to the filter from affecting the delivery of filter-aid.
B. M. Venables.

Filter. J. E. Spalding and R. L. Archer (U.S.P. 1,630,079, 24.5.27. Appl., 20.6.24).—An ascending outlet

conduit leads from the sand bed of the filter to a descending conduit connected to it at the normal level of the liquid within the filter. An adjustable vent admits air above the liquid in the first conduit to prevent draining of the filter through the outlet. H. Holmes.

Purification, decoloration, and deodorisation of liquids and solutions. L'Ultra-Filtre D.M.S. (E.P. 262,131, 26.11.26. Conv., 26.11.25).—The liquid is passed through superposed porous membranes having adsorptive properties, such as nitrocellulose, cellulose acetate, or gelatin. The membranes are divided into groups which can be put into operation independently, the saturated membranes being regenerated by passing through them in a reverse direction the same liquid at a changed temperature, and to which may be added other products or a different liquid. W. G. Carey.

Apparatus for treating air or gas with liquid. Visco Engineering Co., Ltd., and F. C. Smith (E.P. 269,790, 4.10.26).—In a wet scrubber of the type where a number of rotating perforated plates dip into a liquid bath, the perforations are provided with raised edges or lips on the side facing the oncoming gas, with the object of preventing dirty liquid being carried through the perforations. B. M. Venables.

Distributing apparatus. A. Möder (G.P. 428,929, 15.1.25).—Apparatus suitable for treating liquids with gases, for crystallising salt solutions, etc. comprises a number of superposed box-shaped structures of unequal height rotating about a common axis, and provided with helical grooves or channels. L. A. Coles.

Drying and separation from gases of soluble and/or condensable vapours and impurities. Carrier Engineering Co., Ltd., and S. L. Groom (E.P. 269,714, 18.3.26).—The gases (particularly coal gas) are scrubbed with refrigerated water; they preferably pass through two chambers in series, each being provided with water sprayed in opposite directions to the gases from separate refrigerating–washing circuits, and drop arrestors are placed between the two chambers and at the final exit. The refrigerators are preferably of the evaporative type, and a weir, trap, or water seal may be provided for drawing off condensed light liquids, such as benzol, also the excess water due to condensation. B. M. Venables.

Apparatus for the purification of gases. G. König (G.P. 438,833, 7.2.25).—Dust particles are precipitated by passing the gas upwards through a honeycomb filter in which lateral and vertical movements are prevented by vertical or sloping partitions which are intersected by cross-partitions to facilitate the precipitation. A settling chamber may be attached to the filter. L. A. Coles.

Separation of gas mixtures. M. Frankl (F.P. 613,755, 16.1.26. Conv., 16.1.25).—Liquid collectors used in separating gas mixtures by liquefaction are divided into two or more sections, or consist of separate apparatus provided with corrugated sheets of metal, the separate sections being used in rotation. L. A. Coles.

Furnace for supplying drying gases. General Rubber Co., Assees. of D. E. Mansfield (E.P. 267,464, 10.9.26. Conv., 13.3.26).—A combustion chamber is surrounded by a jacket, and both are supplied at the same end with air from a common blower, the air being preferably delivered tangentially into the jacket. The united gases are exhausted either to atmosphere when lighting up and smoky, or to pass over the goods to be dried. The fuel is preferably liquid, and refractory material is used to promote complete combustion. B. M. Venables.

Kiln. Carborundum Co., Ltd. From B. M. Johnson (E.P. 270,520, 6.7.26).—A number of heating chambers each provided with internal "radiating" combustion chambers are interconnected by underground flues, so that air before combustion may be passed over already burnt material, and products of combustion may be used to preheat new material. B. M. Venables.

Refrigerant. G. Barsky, Assr. to American Cyanamid Co. (U.S.P. 1,631,573, 7.6.27. Appl., 5.3.26).—A mixture of monomethylamine and ammonia is claimed. H. Holmes.

Manufacture of an adsorbent. K. Ikeda, H. Isobe, and T. Okazawa, Assrs. to Zaidan Hojin Rikagaku Kenkyujo (U.S.P. 1,630,660, 31.5.27. Appl., 27.12.23).—See E.P. 206,190; B., 1924, 3.

Apparatus for the extraction and recovery of volatile liquids. E. A. Ironside (U.S.P. 1,631,036 31.5.27. Appl., 6.11.25. Conv., 8.11.24).—See E.P. 246,930; B., 1926, 345.

Utilising the heat of materials discharged from furnaces and kilns. Babcock & Wilcox, Ltd. From Babcock & Wilcox Co. (E.P. 269,813, 11.12.26).

Refrigerating apparatus. G. Roos (E.P. 249,873, 24.3.26. Conv., 28.3.25).

[Cover for] hydro-extractors for laundry and like purposes. G. E. Eden (E.P. 270,515, 22.6.26).

Heat-retaining coverings or lagging. J. S. Grimason, and Keasbey-Mattison, Ltd. (E.P. 270,589, 7.12.26).

Gas-purifying plant (G.P. 438,834 and F.P. 614,442).—See XI.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Autoxidation and anti-oxygenic action. XXII. Mode of action of anti-detonators. C. Moureu, C. Dufraisse, and R. Chaux (Chim. et Ind., 1927, 17, 531—535).—Comparison of the action of substances which suppress knocking with the "anti-oxygenic" action described in preceding papers (A., 1926, 581, 1031, 1215), of which the inhibition of the autoxidation of benzaldehyde by traces of quinol and the "poisoning" of certain catalysts may be taken as typical, leads to the conclusion that both actions result from the same cause. None of the various theories of knock suppression which have been proposed up to the present accounts fully for the facts. The theory proposed by the authors is summarised in the equations: $A + O_2 \rightarrow A[O_2]$; $A[O_2] + B \rightarrow A[O] + B[O]$; $A[O] + B[O] \rightarrow A + B + O_2$. A is the oxidisable material or

fuel, and $A[O_2]$ an unstable peroxide which, in the absence of the anti-knock compound B, would pass directly into stable oxidation products. This peroxide, however, in the presence of B interacts with it to form the two peroxides A[O] and B[O], which interact further, in the known manner of antagonistic peroxides, giving A, B, and O_2. Some of the consequences of the theory are briefly discussed. A. B. Manning.

Mechanism of "knock" suppression. H. S. Taylor (Nature, 1927, **119**, 746; cf. Egerton and Gates, A., 1927, 318; B, 1927, 402).—Not only do the lead atoms obtained on thermal decomposition of lead tetraethyl function as oxidation centres, but so also do the free organic radicals producing homogeneous combustion. The effect is supplementary to the inhibitory action of the metal alkyl on the oxidation of the aldehydes produced by partial oxidation of hydrocarbons. A. A. Eldridge.

Active charcoal industry and the oxidation of phosphorus. E. Urbain (Chim. et Ind., 1927, **17**, 536—540; cf. B., 1926, 1003).—The gases produced in the manufacture of active charcoal by the carbonisation of cellulosic material mixed with phosphoric acid contain carbon monoxide and phosphorus vapour. It is not economic to burn these directly; the constituents therefore are separated, the carbon monoxide being used for any suitable purpose, and the phosphorus being oxidised with steam to give phosphoric acid and hydrogen. The latter reaction proceeds conveniently at 400—500° in the presence of active charcoal, which absorbs the phosphoric acid produced; the speed of the reaction is increased by the addition of a trace of a halogen acid. This process is distinct from that described in the patents of Liljenroth (B., 1925, 242; 1926, 320). Since neither the oxidation of carbon monoxide by steam nor the deposition of carbon ($2CO \rightarrow CO_2 + C$) proceeds appreciably under the conditions of the reaction, the mixture of carbon monoxide and phosphorus vapour may be submitted directly to the oxidation process, the resulting gas then corresponding in composition to $CO + H_2$. The disadvantage of using a shaft furnace for the production of phosphorus, namely the low concentration of the latter in the exit gases, can be overcome by absorbing the phosphorus in active charcoal. The slag from this process can be used for cement manufacture. A. B. Manning.

Briquette binder containing residues from alcohol manufacture. W. C. Moore and H. A. Myers (Ind. Eng. Chem., 1927, **19**, 147—149).—Molasses residue from alcohol manufacture, evaporated down to *d* 1·3, will form emulsions with petroleum-pitch asphalt if dust from incinerator flue gases or, better, calcium chloride is used as an emulsifying agent. These emulsions may be employed as a binder in the manufacture of briquettes from anthracite culm. The addition of sulphur to the residue gives a briquette less liable to leaching of the binder, though it does not increase the strength of the briquette. A suitable binder has the formula, in pts. by wt.; concentrated molasses residue, 28; anhydrous sodium carbonate, 9; anhydrous calcium chloride, 10; sulphur, 9; asphalt (60°), 28; and water, 14. The solids are ground and mixed with the residue, added to the asphalt at a temperature somewhat above its m.p., and boiling water is added. The briquettes are generally baked at 315° for 30 min. H. Moore.

Sulphur in coal and coke. W. A. Selvig and A. C. Fieldner (Ind. Eng. Chem., 1927, **19**, 729—733).—From an examination of sixteen samples of coal and coke ranging in sulphur content from about 0·5 to 17% by the Eschka, bomb-washing, and sodium peroxide fusion methods, it is concluded that the last two methods yield results comparable with the standard Eschka method. The three methods are described in detail. The bomb-washing method requires slow and uniform release of the gases after combustion, and thorough washing of the bomb, including valve openings etc. C. O. Harvey.

Design and operation of horizontal retort settings [for producer-gas]. T. H. Goldsmith (Gas J., 1927, **178**, 375—380).—In producer construction, it is recommended that the front wall should slope towards the grate in order to reduce clinker formation above the top steps, whilst for efficient combustion the velocity of producer gas and secondary air entering the combustion chamber should be 9—10 ft./sec. A regenerator is described which allows a uniform speed of travel of the gases throughout its length, a condition necessary for efficient working. Aluminium-coated steel tubes are suggested for use in regenerators at temperatures below 900°. H. D. Greenwood.

Removal of tar fog from coal gas. N. A. Ross (Gas World, 1927, **86**, Coking Sect., 53—55).—The principles of the different methods of removing tar fog are briefly outlined, and a number of arrangements suitable for the recovery of tar fog on the laboratory scale are described. For small quantities, the most efficient combination is a glass-wool extractor, followed by a cotton-wool extractor. A glass-wool diaphragm of 6—8 mm. diam. dealt successfully with the gases (730 litres) from 5 lb. of coal carbonised in a silica retort, collecting 28—30 c.c. of tar. For larger quantities of tar fog, a laboratory modification of the Pelouze and Auduin extractor, followed, if necessary, by a plug of cotton-wool, is recommended. A. B. Manning.

Burning gases in nitrous oxide. H. B. Dixon and W. F. Higgins (Fuel, 1927, **6**, 232—235; cf. A., 1926, 689).—Flames of hydrogen, methane, ethylene, and propylene show a marked increase in size and luminosity, together with the appearance of characteristically coloured zones when the surrounding atmosphere of air is replaced by nitrous oxide. Carbon is deposited from the flame of propylene burning in nitrous oxide, and gradually forms a sheath round the luminous zone. The ignition temperatures of hydrogen, ethylene, and propylene in nitrous oxide have been determined at pressures from 150 to 1000 mm. and times of lag from 0·5 to 5 sec., by the concentric tube method; the values for the three gases at 760 mm. and a lag of 0·5 sec. were 597°, 592°, and 564° respectively. The curves showing the variation of the ignition temperature with pressure exhibit maxima, and are similar to those for ignition in oxygen, the temperatures at the corresponding pressures, however, being always lower in nitrous oxide. Nitrous

oxide alone undergoes no appreciable spontaneous decomposition at temperatures below 700°.

A. B. Manning.

Occurrence of synthol in the process of petroleum synthesis under pressure, and the synthesis from water-gas of paraffin hydrocarbons of high molecular weight. F. Fischer and H. Tropsch (Brennstoff-Chem., 1927, 8, 165—167).—When water-gas is passed over a catalyst comprising finely-divided iron and copper, in the ratio 4 : 1, at 280° and 10—15 atm., a mixture of hydrocarbons and oxygenated compounds is formed. As long as the catalyst is fresh, oily and aqueous products predominate, but after prolonged action a greater quantity of solid paraffins is produced. These adhere to the catalyst, and are extracted with benzene or xylene. After recrystallising, they are colourless and melt in the neighbourhood of 110°. Their mol. wt. is about 1000, and they are of the order of $C_{70}H_{144}$.

W. T. K. Braunholtz.

Action of sulphur monochloride on petroleum hydrocarbons. E. Lorand (Ind. Eng. Chem., 1927, 19, 733—734).—Sulphur monochloride reacts vigorously with unsaturated compounds. With normal paraffin hydrocarbons the reaction is slow even upon heating, but side-chain paraffin hydrocarbons react more readily. The reaction is accompanied by the formation of polymerisation products and the evolution of hydrogen chloride, and it is suggested that, in the case of paraffins, ethylenic linkings are formed, whilst ethylenic compounds yield two hydrogen atoms to form hydrogen chloride with the formation of a triple linking. The reaction might serve as a test for unsaturated compounds.

C. O. Harvey.

Ethyl sulphate in the examination of hydrocarbon oils. J. N. Taylor (Ind. Eng. Chem., 1927, 19, 76—77). —The non-poisonous ethyl sulphate is used to differentiate paraffins and naphthenes from aromatic and unsaturated hydrocarbons, the procedure, though less simple than that with methyl sulphate, being more reliable. 5 c.c. each of mixtures of white oil and benzene to be tested are placed with 5 c.c. of white oil in Egertz tubes, and 16 c.c. of ethyl sulphate are added, the amount of residue being observed at 25°. The results are comparable with the sulphonation residue, with a maximum error of 12%. Dips from low-temperature coal-tars and petroleum oils were tested with concordant results.

H. Moore.

Changes in properties of four unblended mineral oils produced by prolonged treatment with ozone. M. V. Dover and J. H. Cromwell (Ind. Eng. Chem., 1927, 19, 94—96).—Two oils of paraffin and two of asphaltic base were examined before and after treatment with ozone from a laboratory ozoniser at room temperature for 8 hrs., and at 27° for 16 hrs. Lowering of interfacial tension has no direct relation to lowering of the static coefficient of friction, and in some cases the viscosity was inversely proportional to the coefficient of static friction. A more unsaturated oil is generally a more efficient lubricant than a more saturated one.

H. Moore.

Composition of gasoline as indicated by close fractionation. J. B. Hill, L. M. Henderson, and S. W. Ferris (Ind. Eng. Chem., 1927, 19, 128—130).—Four gasolines of various origins were fractionated, and curves prepared showing the relation between the sp. gr. and b.p. of the fractions. Fractionation was conducted in a 5-ft. Snyder column with 47 sections run adiabatically, with a controlled reflux of 5 : 1 at the top, fractions of 2·5% being taken, and also in a 16-ft. column packed with glass rings with electric insulation and controlled reflux, with cuts of 1·2° range. The curves showed regular peaks and troughs, the peaks (high sp. gr. and n values and low aniline point) showing a concentration of the more naphthenic hydrocarbons, and the troughs a higher concentration of the paraffins. All samples have their peaks and troughs in the same places. Re-fractionation caused the troughs to be lowered and the peaks raised.

H. Moore.

Measurement of knock characteristics of gasoline in terms of a standard fuel. G. Edgar (Ind. Eng. Chem., 1927, 19, 145—146).—A standard fuel for the comparison of knocking tendencies of gasolines is made by mixing pure n-heptane, obtained by the distillation of Jeffrey pine oil with a new octane, which is β-*methyl*-β-iso*butylpropane*. A method for preparing this octane from *tert.*-butyl alcohol is given. It has b.p. 99·3°, d 0·6914, viscosity 0·00543 at 18·5°, and n_D^{20} 1·3921. It has knocking tendencies less than those of any commercial gasoline, whilst n-heptane has greater tendency to knock than commercial gasoline. By mixtures varying from 40 to 60% of the two substances respectively, any gasoline may be matched as regards knocking tendencies.

H. Moore.

Bowie-Gavin process for shales. Bowie.—See III.

Pure sulphur from gas sulphur. Gluud, Schönfelder, and Riese.—See VII.

"Spalling" in gas retorts. Rees.—See VIII.

Flames of atomic hydrogen. Langmuir.—See XI.

Erratum.—B., 1927, 435, col. 1, line 33, *for* "G. Varga" *read* "J. Varga."

Patents.

Drying coal and other fuel, or other granular or powdered material. Internat. Combustion, Ltd., and F. H. Rosencrants (E.P. 265,374, 2.2.26. Addn. to E.P. 234,327; B., 1925, 577).—A central drum rotatably mounted on a vertical shaft carries on lugs on its outside a series of superimposed annular plates. These plates have a series of steam-heating pipes cast within them, the steam and drainage connexions being made by two common header pipes carried on the frame of the rotating drum. The material to be dried falls on to the uppermost plate, where it remains for almost one complete revolution of the drum, after which it is scraped on to the next lower plate, and so on to the bottom. Preheated air is admitted to one side of the casing which encloses the rotating member, and, after passage over the material, leaves the casing on the opposite side.

S. Pexton.

Coking coal. H. G. C. Fairweather. From Urbana Coke Corp. (E.P. 269,994, 1.2.26).—A heated fluid is passed through the coal until its temperature is just below the temperature at which it becomes plastic.

The coal is then fused throughout its mass and finally raised to a coking temperature. It may be necessary to maintain the fused condition of the mass in order to permit the escape of volatile matter, the coking temperature being about 750°. The hydrocarbon vapours may be either assisted from the coal by the admission of steam to the carbonisation chamber before the temperature is raised to the coking temperature, or removed merely by the maintenance of a requisite temperature. During the initial heating stages the coal mass may be agitated or remain quiescent. R. A. A. Taylor.

Process and apparatus for coking coal. H. G. C. Fairweather. From Urbana Coke Corp. (E.P. 270,003, 2.1.26).—The coal is heated in two stages—(*a*) at a temperature just below the critical temperature, and (*b*) at a temperature sufficient to complete the coking process. During the first stage only, the fuel is agitated or tumbled, the second being completed rapidly by heating quickly to the temperature of evolution of the hydrocarbon vapours; or the fuel may be heated slowly to the temperature for evolving the vapours. The second stage may be carried out by pouring the coal into a retort previously raised to the requisite temperature. A reservoir may intervene between the first stage and the retort for the second stage, the rate of discharge from it being so regulated as to allow the coal to reach the desired temperature.

R. A. A. Taylor.

Manufacture of briquettes. S. F. Walton (U.S.P. 1,618,248—9, 22.2.27. Appl., 3.12.25).—(A) Wet-ground coal (9 pts.) of predetermined moisture content is mixed with an oil binder (1 pt.) and briquetted. (B) Coal is bonded with an uncarbonised water-soluble sulphide and waterproofed with a cellulosic agent.

S. Pexton.

Manufacture of activated carbon. R. Threlfall (E.P. 270,505, 4.6.26).—Charcoal is heated with sulphur or is exposed at a high temperature to sulphur vapour; the charcoal is subsequently treated to remove the sulphur from it. The charcoal product is washed with an acid, then with water, and finally dried by heat alone or at a high temperature in an atmosphere of hydrogen.

R. A. A. Taylor.

Treatment of wet peat with porous materials. E. von Niessen (G.P. 432,712, 5.10.19).—To remove water from peat the latter is subdivided and mixed with its own weight or more of an incombustible, absorbent material, *e.g.*, pumice, porous pot, etc., in a powdered or small-grained form, and, after the distribution of the water between the peat and the added material has reached an equilibrium, the water is removed by evaporation, the added material being finally separated from the peat by shaking or sieving. A very considerable drying of the peat is thus attained without the use of pressure or other mechanical means.

A. B. Manning.

Production of water-gas and coal-gas from bituminous fuel with simultaneous recovery of ammonia and tar by-products. Power-Gas Corp., Ltd., and N. E. Rambush (E.P. 270,009, 3.2.26).—In a process whereby bituminous fuel is gasified in a shaft generator under conditions which permit of the recovery of by-products, the mixture of coal-gas and water-gas is enriched by carburation by means which do not interfere with the recovery of the by-products. The down-run is effected only through the generator zone, and the gas so made is carburetted; the up-run is effected both through the generator and distillation zones, and the gaseous products treated for recovery of by-products. Two or more generators may work in conjunction with one common carburettor, which may be heated by waste blow-gases successively from each generator. The blow-gases may also be used to raise steam for aiding the distillation of the fuel.

R. A. A. Taylor.

Manufacture of water-gas. N. J. Bowater (E.P. 270,948, 28.9., 1.10., and 9.11.26).—Pulverised fuel is gasified by steam so highly superheated that the formation of carbon dioxide is practically precluded. The heat of combustion during the blow period is returned to the process in the sensible heat of the superheated steam; the sensible heat of the water-gas and surplus steam preheats air for the blow period. The steam is raised in waste-heat boilers by the sensible heat of the gases produced in both blow and make periods. The temperature in the generator is not, during the make period, allowed to fall below that requisite for the immediate combustion of the fuel in the run period. Further heat may be introduced from the generator lining to counter the endothermicity of the water-gas reaction. Completeness of the reaction may be assured by turbulent and counter-flow of the reacting phases, the time and intimacy of contact being so regulated as to produce a product equivalent to the equilibrium product of a lower temperature. R. A. A. Taylor.

Manufacture of carburetted water-gas. A. C. Klein, Assr. to Stone & Webster, Inc. (U.S.P. 1,630,300—1, 31.5.27. Appl., [A], 26.2.21, [B], 18.11.21). —The plant comprises a generator, a carburettor, and a superheater. (A) The fuel bed is heated by an air-blast; the blast gases are excluded from the carburettor, which is simultaneously treated with air to burn out any carbon deposit from the previous gas-making cycle and to heat the carburettor. Steam is introduced through the fuel bed, and the water-gas is then heated and enriched with oil in the carburettor. (B) Air-blasting of fuel occurs in the generator, and the hot blast-gases are passed through the carburettor; steam is then passed into the heated fuel in the generator to form water-gas, which is passed into and enriched in the carburettor; and, finally, the carbon deposit in the carburettor is burned with air, the products of combustion from the generator being excluded. The oil used for carburation is such as will leave a substantial carbon residue. R. A. A. Taylor.

Eliminating carbon monoxide in the exhaust of internal-combustion engines. M. von Wertaur and S. Caplan (E.P. 270,541, 26.8.26. Conv., 19.6.26).—Attached to the exhaust pipe is a shell containing absorbent baffles placed transversely to the path of the gases. The baffles are holders filled with gas-absorbing material, together with a reagent capable of oxidising the carbon monoxide. R. A. A. Taylor.

Separation of tar and ammonia from fuel gases. W. C. Holmes & Co., Ltd., J. Parker, and D. M.

HENSHAW (E.P. 270,852, 5.3.26).—The condensing chamber is situated above and in communication with the vessel into which the condensate is received, both vessels being provided with cooling means. The receiver has outlets at different levels in order that the aqueous and tarry extracts may be drawn off separately. Alternatively, the height of the means for drawing off tar may be adjustable, so that the level of the tar may be varied in the space in which the liquor collects and the liquor drawn off through an outlet provided.

R. A. A. TAYLOR.

Extraction of [aromatic] hydrocarbons from [coal] gas. A. P. BJERREGAARD, Assr. to DOHERTY RESEARCH Co. (U.S.P. 1,624,206, 12.4.27. Appl., 17.1.22).—Coal-gas, freed from tar, ammonia, and pyridine, is washed with mineral oil at about 90° to separate phenolic compounds, and then with mineral oil at ordinary temperature to separate benzene and toluene. Due to the absence of phenolic compounds, no frothing occurs in the subsequent steam-distillation of the mineral oil containing aromatic hydrocarbons. T. S. WHEELER.

Gas-purifying apparatus. DEUTSCHE ERDÖL-A.-G. and K. BOMCKE (E.P. 271,018, 8.3.27).—The dry removal of dust from gas is effected by means of a dust-separating chamber, leading to and from which are several restricted passages through which the gas passes in a horizontal direction at a uniform velocity. These equalising devices consist of small objects, *e.g.*, glass or aluminium beads, shingle, etc., arranged between wire grids, which are so arranged as to offer a minimum of resistance, and are suitably spaced from each other. Each equalising device has a filling port at the top and an emptying port at the bottom, and from one device to the other plates may stretch, the gas passing between them, and the dust collecting upon ribs, waves, etc., which are arranged in staggered relation with each other.

R. A. A. TAYLOR.

Process and apparatus for distilling small samples of coal. R. GEIPERT (E.P. 267,082, 22.9.26. Conv., 4.3.26).—The experimental retort is already heated at the temperature of the test. A sample of coal is introduced in the form of a moulded block or of a number of blocks of regular shape and size, which can be strung together on a wire. The blocks thus occupy the same space in the retort, and accurate comparative tests can be made by successive distillations with similar blocks. The retort is enclosed in an externally-heated heat accumulator, or the retort itself forms a heat accumulator. A gas-tight, refractory tube having one end within the retort and the other outside may be used to introduce the coal and to lead off the gaseous products of distillation. The length of the tube inside the retort should be at least twice the internal diameter of the tube. The tube should be vertical, and have at its outer end a stopper through which passes the wire holding the blocks, the wire being held by a clip so that it can be released for dropping the blocks down the tube. R. A. A. TAYLOR.

Preparation of synthetic petroleum hydrocarbons. E. LEGÉ (F.P. 602,007, 17.11.24).—Coal, alkali or alkaline-earth salts, alkali or alkaline-earth carbonates, and water are allowed to interact under pressure and at the necessary temperature. After being brought into contact with alkali or alkaline-earth chlorides in a further working chamber, the gas mixture produced is allowed to interact at 400—700° in the presence of iron and other metals or metallic oxides as catalysts, then passed over iron or copper at 300—400°, and the products condensed in the solid or liquid form. The residual gases are used for heating purposes. A. B. MANNING.

Fuel for internal-combustion engines. E. G. E. MEYER (E.P. 270,772, 12.11.25).—Hydrocarbon distillates having initial b.p. of 70—80° and final b.p. of 175—225°, and of which not more than 5% distils below 80°, whilst only a relatively small proportion boils above 160°, are rendered suitable for use in internal-combustion engines by the addition of up to 5% of commercial ether, together with a small proportion of a substance adapted to retard the spontaneous evaporation of the ether (*e.g.*, phenol, benzoic acid, ammonia). C. O. HARVEY.

Regeneration of lubricating oils. H. BENSMANN (E.P. 268,284, 16.10.26. Conv., 25.3.26).—In the recovery of used motor oils by removal of colloidally dispersed particles of soot and graphite, and of decomposition (cracking) products from the oil, it is treated at 40—70° with substances which exert a polymerising action on the decomposition products (*e.g.*, stannic chloride, aluminium chloride, sulphuric or phosphoric acid, etc.), whereby asphaltic substances are precipitated and carry down with them the colloidal carbon. The oil passes from a settling tank to a filter containing hydrated aluminium magnesium silicate, which adsorbs organic acids and tarry products. C. O. HARVEY.

Manufacture of a lubricant. A. E. BECKER, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,628,646, 17.5.27. Appl., 21.11.24).—Soap is added to a hydrocarbon oil having lubricating properties, the mixture being then heated at between 176° and the b.p. of the oil. H. ROYAL-DAWSON.

Lubricant. H. H. WILLOCK, S. J. CAPLAN, and J. E. BABB, Assrs. to WAVERLY OIL WORKS Co. (U.S.P. 1,625,969, 26.4.27. Appl., 30.8.21. Renewed 3.9.26).—A lubricant for use at high temperatures comprises petroleum oil, aluminium oleate, oleic acid, and a small proportion of a calcium soap. T. S. WHEELER.

Treating hydrocarbon oils. W. L. COULTAS, JUN. (U.S.P. 1,628,532, 10.5.27. Appl., 12.7.20).—The temperature of the oil is gradually raised by passage through heating coils contained in a series of furnaces, and free hydrogen at a temperature above 760° is introduced into each coil at the point where the oil has reached the vaporisation state. The vapours are fractionated and the high-boiling condensate from each column is re-treated in a second furnace. C. O. HARVEY.

Distilling and cracking hydrocarbon oils. S. L. TINGLEY (U.S.P. 1,627,937—8, 10.5.27. Appl., [A], 12.10.23, [B], 28.12.23).—(A) The oil is heated in a water-jacketed coil contained in a furnace, the water in the jacket flowing in the same direction as the oil, and protecting the latter from direct heat from the furnace. The oil is thus progressively heated and vaporised, and the vapour superheated at the same progressively increasing temperatures as those of the water and the superheated

steam formed. (B) The coil is divided into horizontal sections, and means are provided for the automatic control of the oil level. Prior to heat-treatment, the oil is subjected to radioactive emanations. C. O. Harvey.

Absorption and distillation of hydrocarbons. D. L. Newton (U.S.P. 1,628,055, 10.5.27. Appl., 9.12.25).—Each of a series of superimposed chambers carries a partition holding an absorbing liquid medium, and the gases pass upwards from one chamber to another, the rate of flow of gas into each chamber being controlled independently of the rate of flow into other chambers. C. O. Harvey.

[Cracking] treatment of hydrocarbon oils. C. P. Dubbs, Assr. to Universal Oil Products Co. (U.S.P. 1,628,127, 10.5.27. Appl., 17.12.21).—Oil under pressure passes through a heating zone, and thence to a distilling zone, whence the vapours are withdrawn. The oil supply to the heating zone is controlled by the liquid oil level in the distilling zone, and the oil in the former is kept in circulation. C. O. Harvey.

Treatment of petroleum residue. J. C. Morrell, Assr. to Universal Oil Products Co. (U.S.P. 1,618,669, 22.2.27. Appl., 28.6.26).—The liquid medium is separated from cracked petroleum residue containing suspended coke-like particles, and the residue is rendered homogeneous, so that the suspended particles are stabilised in the mixture with the residual oil, and the product assumes a creamy consistency. S. Pexton.

Extraction and decomposition of bitumen. Deutsche Erdöl A.-G. (G.P. 437,210, 12.11.25).—Bituminous materials, in particular brown coals, are extracted with liquid sulphur dioxide at moderately high temperatures. At 70—80° the whole of the montan wax can be brought into solution, whilst at lower temperatures only the resinous materials can be separated. A. B. Manning.

Recovery of volatile solvents [from gas]. R. Oertel, Assr. to Metallbank u. Metallurgische Ges. A.-G. (U.S.P. 1,631,052, 31.5.27. Appl., 8.6.26. Conv., 7.4.24).—See E.P. 266,145 ; B., 1927, 319.

Purifying gases [from iron carbonyl]. J. Jannek, Assr. to I. G. Farbenind. A.-G. (U.S.P. 1,631,823, 7.6.27. Appl., 8.6.25. Conv., 16.6.24).—See E.P. 247,050 ; B., 1926, 351.

Delivery of pulverised fuel to furnaces, kilns, etc. H. A. Procter (E.P. 265,434, 12.5.26).

Apparatus for cooling coke and the like. G. M. Gill (E.P. 265,337, 10.12.25, and 28.5.26).

Apparatus for receiving coke discharged from retorts used in gas manufacture. F. J. Harrisson and J. W. Drake (E.P. 271,260, 16.6.26).

Controlling gas burners for furnaces. J. Keith & Blackman Co., Ltd., and G. Keith (E.P. 270,786, 5.2. and 19.11.26).

Utilisation of ultra-violet rays for supporting combustion in heat engines and the like. H. A. Gill. From Studienges. für Wirtschaft & Ind.m.b.H. (E.P. 269,808, 9.11.26).

Ammonium sulphate and bisulphate, and sulphur (E.P. 250,990).—See VII.

Resins from hydrocarbons (U.S.P. 1,627,054).—See XIII.

III.—TAR AND TAR PRODUCTS.

The Bowie-Gavin process and its application to the cracking of tars and heavy oils, and to the recovery of oil from sands or shales. C. P. Bowie (U.S. Bur. Mines, Tech. Paper 370, 1926. 42 pp.).—The possibility of cracking tars, heavy oils, etc. without the formation of excessive carbon deposits in the apparatus is claimed. The oil is mixed with varying proportions of inert material such as shale, sawdust, etc. which retains the greater part of the carbon formed. Laboratory experiments showed that on heating heavy oils, tar, etc. with finely-ground shale in a small retort at about 400°, a yield of 75—85% of oil of *d* 0·934 was obtained, the loss consisting of fixed gas and carbon. Preliminary work indicated that on a commercial scale it would be necessary to feed the oil-inert mixture and to extract the inert residue continuously, to maintain the mixture in thin layers to prevent over-heating, and to remove the vapour and any condensate immediately from the retort. A continuous semi-commercial apparatus is described, the throughput being 12 barrels of oil per day. The base of the retort consists of a plate composed of a number of cast-iron hollow segments with tongued and grooved joints, arranged in three concentric rings, the whole being 8 ft. in diameter. Supported on this plate is a wrought-iron cone fitted with a feed pipe and a vapour outlet pipe. Any oil refluxing in the cone is trapped and drained to the vapour outlet. The oil and inert material are mixed externally and, after preheating, fed on to the plate, where the mixture is kept in motion by rabble arms rotated by a central shaft passing through the apex of the cone. The spent material is forced to the periphery of the plate by the rotation of the arms, and falls into a pocket cast in one of the plates, from which it is removed by a spiral conveyor. The retort is heated by hot gases passing through the hollow segments of the plate from the periphery to a central flue below the apparatus. The following results are typical of those obtained from cracking Casmalia crude oil (*d* 0·9935). Oil used, 65 gals. ; shale added, 590 lb. ; oil recovered, 58 gals. (*d* 0·934) = 81% recovery ; gas produced, 2130 cub. ft., calorific value 741—1203 B.Th.U./cub. ft. On distillation, the cracked oil gives gasoline (0—220°) 17%, kerosene (220—275°) 15·5%. The viscosity of the residue above this temperature is such as to render it suitable for fuel purposes. H. D. Greenwood.

Viscosity curve of coal tar and the question of its mathematical validity. H. Mallison and F. Soltau (Brennstoff-Chem., 1927, **8**, 169—173 ; cf. Spiers, J.S.C.I., 1926, **45**, 396 T).—Results obtained with two gasworks tars indicate the impossibility of determining the viscosity at 25° from observations at only two other temperatures, although Spiers' formula affords information if a greater number of different observations are made. In practice, however, a few

viscosity determinations at higher and lower temperatures are sufficient, without the introduction of mathematical formulæ, which cannot be expected to be generally valid for a mixture of the complexity of coal tar. W. T. K. Braunholtz.

Tar fog from gas. Ross.—See II.

Patents.

Tar preparations. Prodorite S.A. (Swiss P. 113,922, 25.2.25).—Tar is treated with a solvent, *e.g.*, "gasöl," heated, with stirring, at 300°, allowed to settle, the solution of heavy tar oils drawn off, and any solvent still remaining in the residue removed by distillation. The residue can be used as cement in the construction of concrete vessels resistant to hot water. A. B. Manning.

Manufacture of fluorescent oil. L. Lilienfeld (U.S.P. 1,625,415, 19.4.27. Appl., 23.7.24. Renewed 30.12.26).—See E.P. 163,271; B., 1922, 50 A.

Tar from gas (E.P. 270,009 and 270, 852).—See II.

Fractional distillation (Austr.P. 104,137).—See II.

Wood preservative (G.P. 435,146).—See IX.

IV.—DYESTUFFS AND INTERMEDIATES.

Patents.

Manufacture of anthraquinone derivatives. O. Y. Imray. From I. G. Farbenind. A.-G. (E.P. 270,840, 25.2.26).—When anthraquinone or one of its homologues is heated below 180° with an arylamine salt having a free *p*-position, diaminodiarylanthrones of the type, $CO<^{C_6H_4}_{C_6H_4}>C(C_6H_4\cdot NH_2)_2$, are obtained. At higher temperatures *meso-pp'*-diaminodiarylanthracenes are formed. 9 : 9-*Bis*-p-*aminophenylanthrone*, m.p. 298°, and 9 : 9-*bis*-(4'-*amino*-3'-*methylphenyl*)*anthrone*, m.p. 255°, are obtained from aniline and *o*-toluidine hydrochlorides, respectively, by heating with anthraquinone at 175° (oil-bath temperature). At 185—190° the products are 9 : 10-*bis*-p-*aminophenylanthracene*, m.p. 300°, and 9 : 10 - *bis*-(4'- *amino* - 3'-*methylphenyl*)*anthracene*, m.p. above 300°. C. Hollins.

Manufacture of anthraquinone derivatives. British Dyestuffs Corp., A. Shepherdson, W. W. Tatum, and H. M. Bunbury (E.P. 270,778, 11.1.26).—Acylated 1 : 4-diaminoanthraquinones (*e.g.*, mono- and di-benzoyl derivatives and the dioxamic acid) are obtained in a single operation by treating leuco-1 : 4-diaminoanthraquinone with acylating agents in nitrobenzene or nitrotoluene, the solvent acting also as an oxidant (cf. E.P. 271,023; following abstract). C. Hollins.

Manufacture of anthraquinone intermediates. British Dyestuffs Corp., A. Shepherdson, W. W. Tatum, and H. M. Bunbury (E.P. 271,023, 11.1.26).—Halogenated 1 : 4-diaminoanthraquinones (*e.g.*, 2 : 3-dichloro-1 : 4-diaminoanthraquinone) are obtained in a single operation by treating leuco-1 : 4-diaminoanthraquinone with halogenating agents in an inert or oxidising solvent (*e.g.*, dichlorobenzene or nitrobenzene). C. Hollins.

Manufacture of anthraquinone intermediates. British Dyestuffs Corp., and W. W. Tatum (E.P. 270,779, 11.1.26).—Leuco-1 : 4-diaminoanthraquinones (*e.g.*, leuco-derivatives of 1 : 4-diamino-, 1 : 4-dimethylamino-, 8-amino-1 : 4-dimethylamino-5-hydroxy-, 1 : 4-dimethylamino-5-hydroxy-, and 1 : 4-dimethylamino-5 : 6-dihydroxy-anthraquinones) are oxidised by heating a salt (*e.g.*, the hydrochloride) of the leuco-base in an oxidising solvent (*e.g.*, nitrobenzene) or in an inert solvent with an added oxidant. C. Hollins.

Manufacture and application of new dyes. Soc. Chem. Ind. in Basle (Ges. für Chem. Ind. in Basel) (E.P. 258,854, 8.9.26. Conv., 24.9.25).—Dyes, especially suitable for chroming on wool, are obtained by the action of formaldehyde and sodium bisulphite on the azo dyes formed by coupling diazotised 1-amino-2-naphthol-4-sulphonic acid with α- or β-naphthol. Wool is dyed from a formic or acetic acid bath, and on chroming gives deep black shades. C. Hollins.

Manufacture of alkylnaphthalenes chlorinated in the nucleus. I. G. Farbenind. A.-G. (E.P. 263,844, 29.12.26. Conv., 29.12.25).—Alkylnaphthalenes are chlorinated in the presence or absence of a solvent such as ether or carbon tetrachloride by treatment with sulphuryl chloride. 1-*Chloro-4-methylnaphthalene*, b.p. 278—283°, 1-*chloro-2-methylnaphthalene*, b.p. 273—275°, a *monochloro*-2 : 6-*dimethylnaphthalene*, m.p. 39°, b.p. 293—297°, and a *dichloro*-2 : 6-*dimethylnaphthalene*, m.p. 135°, b.p. 328—333°, are described. C. Hollins.

New azo dyes and process of dyeing cellulose acetate. British Dyestuffs Corp., J. Baddiley, and J. Hill (E.P. 270,428, 12.2.26).—Brown shades fast to light and washing are obtained on cellulose acetate materials by the use of insoluble disazo dyes of the type : nitroarylamine → amine → amine of the benzene series. Suitable first components are *m*- and *p*-nitroanilines, 2 : 4-dinitroaniline, picramic acid, 4 : 5-dinitro-α-naphthylamine, etc. The middle component may be aniline, *m*-toluidine, cresidine, α-naphthylamine, 1-amino-2-naphthyl ethyl ether, etc. Suitable end components are *m*-phenylenediamine, *m*-toluidine, cresidine, and the like. Sulphonic groups must be absent. The dyeing may be done from an aqueous suspension in the presence of a dispersing agent. C. Hollins.

Manufacture of new azo dyes and process of dyeing. British Dyestuffs Corp., J. Baddiley, P. Chorley, and R. Brightman (E.P. 270,446, 24.2, and 8.4.26).—Azo dyes obtained by coupling diazo compounds, excepting diazotised aminonaphthols and their derivatives, with 8 : 8'-dihydroxy-2 : 2'-dinaphthylamine-6 : 6'-disulphonic or -3 : 3' : 6 : 6'-tetrasulphonic acid ("di-γ-acid" or "di-2R-acid") give level dyeings on viscose and other regenerated cellulose silks. In suitable cases the dye may be diazotised on the fibre and developed, or may be coupled on the fibre with diazo components. Thirteen examples are given, the shades being brown to violet, blue, and black. C. Hollins.

Separation of aromatic amines (E.P. 270,930).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Effect of partial hydrolysis on the alkali solubility of wood. L. F. Hawley and W. G. Campbell (Ind. Eng. Chem., 1927, 19, 742—744).—Sitka spruce

sawdust was treated with varying concentrations of hydrochloric acid (0·5—15·0%) for 6 hrs. at 100°, and the product analysed both before and after treatment with 1% sodium hydroxide solution at 100° for 1 hr. Whilst the original wood could be completely accounted for by determinations of cellulose, lignin, " pentosans not in cellulose " (*i.e.*, total pentosans — pentosans in crude cellulose) and water-soluble (total 102%), this was not so in the case of the partially hydrolysed products (total 93—98%). The latter probably contain a cellulose degradation product which does not appear in the above determinations, and is not present in the original wood. The solubility of cellulose in 1% sodium hydroxide at 100° was considerably increased by acid hydrolysis, whilst that of lignin was only slightly affected. Very mild hydrolysis, which had little influence on cellulose, considerably increased the alkali-solubility of the pentosans present. The total alkali-solubility of partly decayed wood was much greater than that of wood which had been hydrolysed to the same extent, *i.e.*, had suffered equal loss in weight. W. J. Powell.

Constants of cellulose pulp. Change with time of cooking. J. Chintschin (Zellstoff u. Papier, 1927, 7, 235).—Prolonged boiling of pulp results in decrease of its lignin content, suppression of the bromine number or chlorine factor, decrease in the quantity of active chlorine absorbed in bleaching, and increase in the amount of the fibre stained by Congo Red. The copper number (Schandrock and Häen–Low) increases, and correspondingly the resistance to baryta (which refers to increase in the quantity of cellulose decomposition products) decreases. Decrease of the total sulphur dioxide content of the cooking liquor apparently produces an increase in the pentosan content. Fluctuations in the α-cellulose content show no definite relation to the duration of boiling, concentration of the liquor being apparently a more important factor, but definite conclusions cannot be drawn owing to the insignificant differences in the amounts of α-cellulose. The duration of boiling exerts a proportionally smaller action on the decrease of the resin content, whilst the amount of ash is also decreased by prolonged cooking. B. P. Ridge.

Pulping of pine wood by the sulphite process. C. G. Schwalbe and K. Berndt (Cellulosechem., 1927, 8, 66—68).—A criticism of Hägglund's conclusions (B., 1927, 294). The latter's own results show that pine wood after extraction with benzene or ether alone cannot be successfully pulped by the sulphite process, even under the conditions used in his sealed-tube experiments, which differed widely from those obtaining on the large scale in regard to such factors as time of boiling, means of agitation, acid concentration, maximum pressure, and size of chips. His conclusion that the age of the wood has no effect on the pulping properties is not valid for the same reason. W. J. Powell.

Recovery of solvents. Wiesenthal.—See I.

Nitrocellulose for lacquers. von Mühlendahl and Schulz.—See XIII.

Sulphite extract as tanning material. Wallace and Bowker.—See XV.

Patents.

Manufacture of artificial silk. F. C. Niederhauser and A. E. Sunderland, Assrs. to Viscose Co. (U.S.P. 1,625,562, 19.4.27. Appl., 9.11.20).—In the manufacture of viscose silk, the addition of glue (2%) to the sulphuric acid setting bath restricts the deleterious action of the acid on the filament and inhibits the crystallisation of any salt left in the filament after removal from the bath. T. S. Wheeler.

Preparation of hollow artificial textile fibres from viscose. British Enka Artifical Silk Co., Ltd. From N. V. Nederlandsche Kunstzijdefabr. (E.P. 253,477, 5.3.26).—Solutions of low viscosity, such that when tested by the steel ball method the time of descent is 3 sec. or less (steel ball ⅛ in. diameter, length of tube 8 in.) are spun, without degasification, into a bath which precipitates the viscose quickly. The presence of a zinc salt (*e.g.*, 2% of zinc sulphate) in this bath is an advantage, and a finely-powdered solid (*e.g.*, pumice) may be mixed with the viscose before spinning in order to promote the formation of gas bubbles in the fibres. B. P. Ridge.

Manufacture of plastics. L. Lilienfeld (U.S.P. 1,625,416, 19.4.27. Appl., 9.1.26. Conv., 15.5.20).—The products described in E.P. 163,271 (B., 1922, 50 a) are of value as plasticising agents for cellulose ethers. T. S. Wheeler.

Composition for use in making sulphate or kraft pulp. H. B. Kipper (U.S.P. 1,629,393, 17.5.27. Appl., 26.11.24).—Sodium hydrogen sulphate mixed with sufficient sodium carbonate to give sodium sulphate, is added to black liquor from the sulphate-cellulose process before evaporation, to make up losses in sodium sulphide during the process. T. S. Wheeler.

Insulating material for electric condensers and the like. J. Lahousse (E.P. 251,970, 27.4.26. Conv., 8.5.25).—A dielectric for condensers consists of paper etc. impregnated with compounds of the amide series solid at ordinary temperatures, but fusible at conveniently low temperatures, particularly urethanes, anilides, carbamides, their sulpho- and chloro-derivatives. By adding to the amide a small quantity of a second substance, the two being reciprocally soluble, a sufficient amount of eutectic (m.p. 30—70°) is formed at the end of cooling, acting as a binding agent, and thus avoiding the detrimental effects of a break in the dielectric mass on solidification. S. S. Woolf.

Production of insulating paper containing phenol resins. C. A. Haanen, Assr. to Felton & Guilleaume Carlswerk A.-G. (U.S.P. 1,630,424, 31.5.27. Appl., 27.11.26. Conv., 29.4.26).—Phenol resin solutions are added to fibrous pulp in which the water has been replaced by solvents for phenol resins, and the resin is precipitated in and on the fibres by the addition of water. The mass is worked up to form paper and pressed articles. S. S. Woolf.

Treatment of fibres for spinning. L. Ubbelohde (U.S.P. 1,629,241, 17.5.27. Appl., 26.6.25. Conv., 23.2.25).—See E.P. 254,357; B., 1926, 782.

Manufacture of artificial silk or the like. J. O. Zdanowich (U.S.P. 1,630,285, 31.5.27. Appl., 12.8.26. Conv., 6.5.25).—See E.P. 260,642; B., 1927, 103.

Machine for grinding paper pulp. K. G. V. Sörbom (E.P. 262,070, 27.10.26. Conv., 27.11.25).

Filtration (E.P. 270,461).—See I.

Dyeing cellulose acetate (E.P. 270,428).—See IV.

Dyeing process (E.P. 270,446).—See IV.

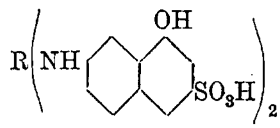
Ligno-tanning materials (U.S.P. 1,629,448).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Chlorine in bleach liquors. Hausner.—See VII.

Patents.

Dyeing artificial silk. British Dyestuffs Corp., J. Baddiley, P. Chorley, and R. Brightman (E.P. 270,883, 16.4. and 11.9.26).—Regenerated cellulose (*e.g.*, viscose) silks are dyed in level shades by azo dyes made by coupling suitable diazo components (1 or 2 mols.) with compounds of the type:



in which R represents a carbonyl or thiocarbonyl group or a straight chain made up of two or more methylene and/or carbonyl groups. Suitable diazo components are aniline, its homologues and derivatives (including carboxylic and sulphonic acids), naphthylamines and their sulphonic acids. 23 examples (reds, violets, and browns) are given, the coupling components being 8 : 8′-dihydroxy-2 : 2′-dinaphthylcarbamide-6 : 6′-disulphonic acid, the corresponding thiocarbamide, *NN*′-8 : 8′-dihydroxy-6 : 6′-disulpho-2 : 2′-dinaphthylethylenediamine (R = ·CH : CH·), *NN*′-8 : 8′-dihydroxy-6 : 6′-disulpho-2 : 2′-dinaphthylglycinamide (R = ·CO·CH_2·), 8 : 8′-dihydroxy-2 : 2′-dinaphthyloxamide-6 : 6′-disulphonic acid (R = ·CO·CO·), and *NN*′-8 : 8′-dihydroxy-6 : 6′-disulpho-2 : 2′-dinaphthyl-$\alpha\gamma$-diaminoacetone (R = ·CH_2·CO·CH_2·). In suitable cases the dyes may be developed or coupled on the fibre. C. Hollins.

Dyeing mixed textiles. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 247,224, 8.2.26. Conv., 7.2.25).—Wool and silk in mixtures are equally dyed in level yellow to orange shades by pyrazolone dyes containing one or more carboxylic ester groups in the diazo or the coupling components or in both. Only such sulphonic or free carboxylic groups as are necessary to give the requisite solubility should be present. Examples are: *o*-chloroaniline $\rightarrow$ ethyl 1-*p*-sulphophenyl-5-pyrazolone-3-carboxylate; ethyl anthranilate $\rightarrow$ 1-*p*-sulphophenyl-3-methyl-5-pyrazolone; ethyl *m*-aminobenzoate $\rightarrow$ ethyl 1-*p*-sulphophenyl-5-pyrazolone-3-carboxylate. The preparation of ethyl 1-(6′-sulpho-2′ : 4′-dimethylphenyl)-5-pyrazolone-3-carboxylate from *m*-4-xylidine-5-sulphonic acid is described. C. Hollins.

Finishing, mercerising, and ornamenting textiles. E. Hausheer (E.P. 266,466, 24.8.25).—The material is treated wholly or locally with a solution containing 8—18% of sodium hydroxide as basis, with or without the addition of 0·1% or more of natural silk, other protein, or agar-agar, and/or up to 5% of dissolved cellulosic material which may also contain pigments or powdered metal. It is then subjected to the action of ammonia, carbamide or its derivatives, washed with acid and water, and dried. Alternatively, 2—10% of ammonia is added to the sodium hydroxide, the material treated therewith, mangled down to about twice its original weight, subjected in the tensioned state for several minutes to a temperature of − 7° to − 12°, and then treated with a 5% sulphuric acid solution, washed, and dried; or the material impregnated with sodium hydroxide is exposed to an ammonia atmosphere at a temperature of − 7° to − 15°. Before acid washing, the excess of the impregnating agent is removed by brushing, mangling, or scraping, treated yarns being passed through circular holes, corresponding to the thread diameter, in divided, hinged bars. Tensioning of the material may be regulated to produce various effects. B. P. Ridge.

Application of dyes (E.P. 258,854).—See IV.

Algin compounds (U.S.P. 1,625,301).—See XX.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Preparation of sulphuric acid from gypsum. H. Molitor (Chem.-Ztg., 1927, **51**, 329—332, 370—373).—Details are given of the important deposits of gypsum and anhydrite on the Moselle followed by an account of the various methods which have been proposed to utilise them as a source of sulphuric acid. The Bambach process effects the decomposition of gypsum at 1400° by means of the surface combustion of producer gas with air on the gypsum itself, which is crushed to small pieces, but not powdered. Alternatively, the air may be shut off at intervals so as to allow the alternate formation and decomposition of calcium sulphide. The principal difficulty is the tendency to slagging, due to the closeness of the decomposition temperature to the m.p. of calcium sulphate, and the risk of formation of basic sulphate or sulphite of lower m.p. Anhydrite is more difficult to work than gypsum. The quicklime produced is "dead-burnt." With care, however, the process can be carried out successfully. Basset heats gypsum in a rotary furnace with clay and coal, steam being blown in, producing cement as residue. The Bayer process, by which 2800 tons of sulphur trioxide are produced monthly at Leverkusen, uses powdered anhydrite and waste calcium sulphate. This is mixed with powdered shale and coke, and fired with powdered coal in rotary cement kilns, in a slightly oxidising atmosphere. The gas contains 6—7% SO_2. The B.A.S.F. have given large-scale trials to a process in which gypsum is allowed to interact with magnesia and carbon dioxide, whereby it is partly converted into magnesium sulphate. The latter is decomposed by heating with powdered coke at 450—500°. The calcium carbonate produced as a by-product is difficult to dispose of. This difficulty also arises with the process operated by the same firm on a large scale in which the magnesia is replaced by ammonia. In the Bambach process the carbon dioxide is replaced by sulphur dioxide. In this case the by-product is calcium sulphite, which is either sold as such, or converted into sulphate by interaction with sodium hydrogen sulphate, sulphur dioxide being also liberated. The main reaction may be carried out in towers or in a series of closed iron cylinders,

and is best performed at the b.p. of the solution. The second step is chiefly of interest as a means of utilising the acid content of nitre-cake and converting the residue into saleable saltcake. The Badische process for recovering elementary sulphur from gypsum has been abandoned owing to the poor yield. It has also been proposed to recover elementary sulphur by reducing gypsum with methane, or by reducing to calcium sulphide, decomposing with magnesium chloride solution, and proceeding as in the Claus process. C. Irwin.

Production of pure sulphur from gas sulphur. W. Gluud, R. Schönfelder, and W. Riese (Brennstoff-Chem., 1927, 8, 168—169).—Crude sulphur, recovered from coal-gas by the method of the Ges. für Kohlentechnik (cf. B., 1927, 321) may be purified by distillation, although difficulties are met with when it contains much tarry matter or when large quantities have to be dealt with. An alternative method is to burn part of the sulphur, absorbing the sulphur dioxide in concentrated ammonia liquor, and to dissolve a further part of the crude sulphur in the ammonium sulphite solution so obtained. From the resulting ammonium thiosulphate solution pure sulphur is precipitated by adding sulphuric acid, and the liquor, after centrifuging, is worked up for ammonium sulphate. W. T. K. Braunholtz.

Determination of available chlorine in bleach-liquor. J. Hausner (Chem.-Ztg., 1927, 51, 373—374).—A method intended for unskilled control. A stoppered measuring cylinder is filled with the sample to be tested up to a zero mark. A solution containing 0·85 g. of crystalline sodium thiosulphate and 0·14 g. of sodium indigodisulphonate per litre is run in until the colour changes to greenish-blue. The percentage of available chlorine is then read direct from a graduation. The standard solution, in concentrated form, remains unchanged for upwards of five months, and the method is reasonably accurate. C. Irwin.

Manganese interference in the *o*-tolidine test for available chlorine. E. S. Hopkins (Ind. Eng. Chem., 1927, 19, 744—746).—In the presence of easily reducible salts of manganese and other metals the results of the *o*-tolidine test for free chlorine are unreliable, since a yellow colour similar to that due to chlorine is produced; *e.g.*, 0·01 pt. of potassium permanganate per million produces a colour intensity equal to that of 0·03 pt. of chlorine per million. Stable salts of manganese such as the sulphate or chloride at a concentration as high as 100 pts. per million do not produce a colour, but manganous hydroxide in the presence of air readily yields a positive result. The test is unsuitable for a determination of residual chlorine in water supplies containing manganese. W. J. Powell.

Charcoal and phosphorus. Urbain.—See II.

Barytes in rubber industry. Dawson and Hartshorne.—See XIV.

Patents.

Manufacture of sulphuric acid. H. Petersen (E.P. 244,764, 270,988—9, [A], 11.12.25, [B, C], 16.12.26. Conv., [A], 16.12.24, [B], 12.10.26).—(A) Gases containing sulphur dioxide are treated in reaction enclosures filled with packing material and irrigated with solutions of nitrosylsulphuric acid in high-grade sulphuric acid containing such an amount of nitrogen oxides that the sulphur dioxide is in excess. A fine jet of water is introduced in small quantity, so that the resulting nitrous sulphuric acid has the same nitrous content as the acid fed to the same reaction enclosure. The acid is denitrated in a tower in front of the system. (B) Excess of water in sulphurous gases is removed by treatment with acid (*d* 1·71) withdrawn from the circulating acid in a tower in front of the system, the resulting weak acid being returned to the plant instead of water for acid formation. (C) Sulphur dioxide gases containing excess of moisture are divided into two portions, one of which effects the denitration of the commercial acid by intimate contact of acid and gas in a tower packed with inert material in grains of not more than 40 mm. diameter, the other portion being introduced into the plant at points where water is necessary. W. G. Carey.

Sulphuric acid chambers. Packards & J. Fison (Thetford), Ltd., and R. T. Maudsley (E.P. 270,826, 17.2.26).—Externally water-cooled chambers of the Mills–Packard type are flexibly supported by a series of links pivoted at one end to a vertical lead lug burnt on to the chamber from top to bottom, and at the other on to brackets secured to the framework of the chamber, the brackets having adjustable eyebolts. W. G. Carey.

Distillation of hydrochloric acid. L. C. Drefahl, Assr. to Grasselli Chemical Co. (U.S.P. 1,628,829, 17.5.27. Appl., 5.4.23).—A continuous stream of crude hydrochloric acid of greater strength than the constant-boiling acid flows over a heated surface in a thin layer, the vapours being condensed and withdrawn; the rate of flow is regulated according to the rate of vaporisation. H. Royal-Dawson.

Manufacture of acetic acid. H. Dreyfus (E.P. 268,845, 7.11.25).—Methyl alcohol and carbon monoxide at atmospheric pressure are heated below 400—500°, and preferably at 200—350°, with catalysts capable of forming acetates which split off acetic acid at temperatures below 400—450°, and preferably at 200—300° (cf. E.P. 264,558; B., 1927, 268). Such catalysts are copper oxide or acetate, zinc oxide, acetate, or methoxide, and finely-divided zinc, aluminium, or copper, or mixtures of any of these with potassium acetate or sodium acetate. W. G. Carey.

Production of phosphoric acid. W. H. Waggaman and H. W. Easterwood, Assrs. to Victor Chemical Works (U.S.P. 1,630,283, 31.5.27. Appl., 23.1.24).—A mixture of phosphate rock, silica, and coke is heated with solid fuel, and oxidising gases are introduced at one end of the mass to burn the fuel, while additional oxidising gases are introduced at various points to oxidise the phosphorus evolved. W. G. Carey.

Synthetic production of ammonia. F. Uhde (E.P. 247,225 and 259,230, [A] 8.2.26, [B] 30.9.26. Conv., [A] 9.2.25, [B] 2.10.25).—(A) Catalysts entirely free from water for the production of ammonia from its elements at about 425° are prepared from ferro- or ferri-cyanides and metallic salts of the iron group by solution or suspension in water-free polyhydric alcohols, *e.g.*, glycol. (B) Superheating of the catalyst is obviated

by conveying the gases through double pipes arranged in the contact chamber in a double counter-current, so that the fresh gases enter through the inner pipes in the same direction in which they are conducted through the catalyst, and are then led through the outer pipes in counter-current to the entering and to the reacting gas current, and thence to the contact mass for reaction. The diameters of the pipes are calculated so that the heat transference assures a practically uniform temperature throughout the whole contact chamber. W. G. Carey.

Simultaneous manufacture of ammonium sulphate and bisulphate and of sulphur. C. Harnist (E.P. 250,990, 20.4.26).—Solutions of ammonium salts of one or several oxygenated acids of sulphur are heated under pressure. If bisulphite or normal sulphite is used risk of explosion is prevented by the direct introduction of steam, which starts the reaction and agitates the liquid. Solutions to be so treated are obtained from ammonia and hydrogen sulphide gases from coal distillation by absorption, while hot and before tar removal in water, in saturators or scrubbers and treatment with sulphur dioxide. The decomposition of hydrogen sulphide by sulphur dioxide is accelerated by the action of a strong electric field. W. G. Carey.

Manufacture of ferric oxide. D. G. Zalocostas (U.S.P. 1,630,881, 31.5.27. Appl., 28.3.25. Conv., 16.4.24).—Ferrous sulphate crystals are passed through a dehydrating chamber having gas burners which project flames over the surface of the crystals and are so regulated that the water of crystallisation is vaporised as it is liberated, a temperature of 64° being maintained in the chamber. The dehydrated product is ground, oxidised, and roasted. W. G. Carey.

Manufacture of chromic chloride. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 269,028, 26.7.26).—Anhydrous chromic chloride is made by treating ferrochromium with chlorine, with or without a small quantity of a reducing agent, *e.g.*, carbon monoxide, in a heat-insulated revolving furnace. Heat is necessary only to start the reaction; thereafter the temperature is controlled by varying the amount of chlorine so that the iron chloride formed is sublimed. W. G. Carey.

Manufacture of hydrated chromic chloride. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 271,016, 26.7.26).—Anhydrous chromic chloride is converted into the hydrated salt without the aid of reducing agents by exposing an aqueous suspension to the action of a cathodically polarised conductor, *e.g.*, in a lead-lined iron vessel having a chromium anode and using a very low current density. The heat generated in the process enables a highly concentrated solution to be produced, which is filtered while hot and solidifies on cooling. W. G. Carey.

Production of high-acid phosphates. E. C. R. Marks. From Parker Rust Proof Co. (E.P. 270,820, 16.2.26).—The dihydrogen orthophosphate of manganese, zinc, cadmium, or iron is made by dissolving the metal in a 60—70% solution of orthophosphoric acid at 100—115°, filtering, cooling, and crystallising, finally drying under non-oxidising conditions below 60°. Ferromanganese is used if the manganese and iron salts are required together. W. G. Carey.

Manufacture of barium salts, especially the carbonate, free from sulphur. F. Falco (E.P. 270,559, 5.10.26).—Barium carbonate, precipitated from barium sulphide by carbon dioxide, is mixed into a paste with a reducing agent which decomposes and volatilises on heating, such as a 2% solution of formic or oxalic acid, and is heated in a rotary drum to drive off the hydrogen sulphide and sulphur dioxide and to decompose the reducing substance. Barium salts may be treated in solution by the same reducing agents, acidifying with the appropriate acid to liberate the sulphur compounds. W. G. Carey.

Continuous purification of crude carbon disulphide. E. Legeler and P. Esselmann (E.P. 254,676, 1.12.25. Conv., 6.7.25. Addn. to E.P. 238,489; B., 1925, 803).—Crude carbon disulphide is run through the inner part of a double concentric rectifying column filled with Raschig rings and surrounded with a heating jacket at the bottom. From the inner part of the column hydrogen sulphide is carried off, and from the outer part carbon disulphide free from hydrogen sulphide is distilled from the upper end, while the concentrated solution of sulphur in carbon disulphide remaining is continually withdrawn for further separating treatment. W. G. Carey.

Method of chlorinating solutions. J. H. MacMahon, Assr. to Mathieson Alkali Works, Inc. (U.S.P. 1,616,134, 1.2.27. Appl., 9.7.25).—The liquid is circulated by a pump and the pressure is increased on the discharge side of the pump by restricting the flow, chlorine being introduced in the region of increased pressure. W. G. Carey.

Manufacture of phosphorus acids and hydrogen. I. G. Farbenind. A.-G. (E.P. 262,447, 2.12.26. Conv., 3.12.25).—Heat produced by the interaction of phosphorus and steam is recovered by separating the phosphorus acids formed from the reaction mixture by a water-operated condenser maintained at 100° or over, the condenser generating steam at atmospheric or higher pressure. The excess of steam and hydrogen from the reaction passes through a second condenser and generates steam below atmospheric pressure, which is compressed and superheated for use in the process. W. G. Carey.

Purification of burner gases and manufacture of chemically-pure sulphuric acid. M. Krafft (U.S.P. 1,627,977, 10.5.27. Appl., 25.6.23. Conv., 30.6.22).—See E.P. 220,413; B., 1924, 868.

Production of hydrocyanic acid. R. Koepp & Co., Assees. of G. Bredig and E. Elöd (U.S.P. 1,627,144, 3.5.27. Appl., 21.11.24. Conv., 7.12.22).—See E.P. 229,774 and 229,973; B., 1925, 315.

Ammonia from gas (E.P. 270,009 and 270,852).—See II.

Depilatory (G.P. 436,149).—See XV.

VIII.—GLASS; CERAMICS.

Crystallisation of glass. A. Lecrenier (Bull. Soc. chim. Belg., 1927, 36, 137—148).—A rare example of crystallisation is furnished by a specimen of glass in the collection of the Institut Chimique at Nancy. Analysis

of the glass yielded the following numbers: SiO_2, 75·54%; PbO, 0·37%; $Fe_2O_3 + Al_2O_3$, 0·35%; Mn_3O_4, 0·25%; CaO, 7·51%; MgO, 0·18%; Na_2O, 14·51%; SO_3, 0·43%; Sb_2O_3, traces; total, 99·16%. Spherolites of varying size, the majority, however, being about 4—5 mm. in diameter, are formed within the material, and their number increases from year to year. The spherolites have the same composition as that of the surrounding glass. The crystalline phase appears to be cristobalite. On allowing a fused barium crown leger glass (SiO_2, 48·08%; As_2O_3, 0·57%; BaO, 29·83%; Al_2O_3, 1·65%; ZnO, 8·09%; K_2O, 6·87%; Na_2O, 1·36%; B_2O_3, 3·55%) to cool, a large number of spherolites of devitrification, about 5—7 mm. in diameter, were formed within the material. As in the preceding case, the spherolites have the same composition as the surrounding glass. Devitrification was not accentuated by heating at 600°, but was completed on raising the temperature to 700°. The crystalline phase appears to be the silicate, $BaSi_2O_5$. No well-defined crystal which could possibly have acted as a centre of crystallisation was found in any of the spherolites. Crystal soda glass (Na_2O, PbO, SiO_2) decomposes on cooling with separation of cristobalite. Glasses containing cuprous oxide crystallise to form a vitreous, opaque mass having a characteristic reddish-brown colour. The crystalline phases are cristobalite and tridymite. Wollastonite has been found in numerous specimens of plate glass.

J. S. CARTER.

Ionometric measurement of the acidity and alkalinity of glasses by means of the Luers quinhydrone potentiometer. A. MAURI (Giorn. Chim. Ind. Appl., 1927, **9**, 168—169).—Tests of the alkalinity, similar to those already carried out by Clark's colorimetric method and by the potentiometric method with hydrogen, have been made on Muranese (Tenax), Neapolitan (Crist. Naz.), and Jena (Fiolax) glasses. The p_H values of the solutions obtained by treating these glasses with water of p_H 5·43 in the cold (at 134° for 1 hr.) are, in the above order, 5·65 (5·45), 4·74 (5·62), and 5·75 (6·33). These results differ in absolute magnitude from, but are parallel to, those previously obtained.

T. H. POPE.

"Spalling" in silica gas retorts. W. J. REES (Trans. Ceram. Soc., 1926, **25**, 321—325).—Normally there is little or no penetration or deposition of carbon within silica retort material, but in the two cases investigated several small patches of carbon were visible on the internal face of the "spalled" pieces. Faint cracks were detected round these small black patches. Analyses showed that the black patches contained 12·5% C and 5·18% Fe_2O_3, whereas the normal material contained no carbon and only 0·92% Fe_2O_3. It is suggested that small segregations of iron oxide in the silica retort material have acted as nuclei for the deposition of carbon. This has caused a weakening of the silica material round the deposited carbon, with the gradual development of cracks. In a laboratory test, "carbonised" iron oxide spots were obtained by subjecting silica brick impregnated with iron oxide to the action of a current of carbon monoxide at 700—900°.

A. T. GREEN.

Discoloration of clays during firing. J. KONARZEWSKI and A. E. J. VICKERS (Trans. Ceram. Soc., 1927, **26**, 1—20).—The influence of water vapour and sulphur dioxide upon the colour of fired ware was investigated. Six different clays were tested; they were fired in an electric tube furnace through which air, water vapour, and sulphur dioxide, or a mixture of these, could be passed at will. Two series of experiments were made, the firing period in the first being 7½ hrs. and in the second 10 hrs. The colour of the fired clays was measured by means of the Lovibond tintometer. It was observed that the presence of water vapour during the dehydration and oxidation periods of firing, and the presence of sulphur dioxide during the whole time of firing, produced a reddish coloration of white and yellow burning clays. The presence of water vapour during the last stage of firing produced a lighter colour than was obtained under normal conditions. The action of the gases on clays is strongest between 500° and 600°. The discoloration may be removed more or less by prolonged firing in an oxidising atmosphere. The sulphur dioxide seemed to be adsorbed by the clays, and the presence of water vapour retarded the process of dehydration, which then took place more suddenly and at a higher temperature, causing more complete breakdown of the iron compounds.

F. SALT.

PATENTS.

Manufacture of glass. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY & CIREY (E.P. 264,495, 11.1.27. Conv., 12.1.26).—In glasses used for absorbing *X*-rays and rays of similar short wavelength, and containing more than 50% of lead oxide or 20—50% of lead oxide with 40—10% of barium oxide, zirconium oxide, up to 10%, is employed to replace some of the silica. The rate of setting of the glass on cooling is thereby increased, whilst the glass also possesses increased opacity to the rays, and is less sensitive to exterior agents. Batch materials used are zirconia or zircon.

A. COUSEN.

Manufacture of vitreous silica. L. B. MILLER, Assr. to GENERAL ELECTRIC CO. (U.S.P. 1,628,468, 10.5.27. Appl., 29.12.26).—Vitreous silica is prepared from crystal quartz by heating at a temperature but little below 1700°, then cooling to below 270°, and finally fusing *in vacuo*.

A. COUSEN.

Glass composition. W. F. BLEECKER (U.S.P. 1,629,648, 24.5.27. Appl., 22.7.22).—A mixture consisting of 75 pts. of sand, 80 pts. of soda ash, 70 pts. of litharge, 20 pts. of borax, 30 pts. of nitre, and 4—24 pts. of uranium oxide is used, and gives a transparent henna-coloured lead glass.

H. ROYAL-DAWSON.

Glass. F. M. LOCKE (U.S.P. 1,626,042, 26.4.27. Appl., 28.8.16. Renewed 29.10.26).—A borosilicate glass with a low coefficient of expansion contains 70—79% of silica, not above 28% of boron trioxide, not above 5% of alumina, and not above 9% of alkali oxides, which latter are added to the batch as nitrates prior to fusion.

T. S. WHEELER.

Manufacture of synthetic aquamarine stones. J. F. RIERA (E.P. 271,316, 4.10.26).—A mixture of silica and hydrated or anhydrous beryllia is fused with alumina

and colouring matter containing cobalt nitrate, together with a flux, *e.g.*, lithium carbonate or borax. The fused product is broken into fragments, softened by heat, and moulded under pressure to the desired shape before polishing. B. W. Clarke.

Manufacture of a ceramic product. T. C. and W. O. Prouty, Assrs. to American Encaustic Tiling Co., Ltd. (U.S.P. 1,628,910, 17.5.27. Appl., 7.3.25).—A bisque of talc, clay, and calcium sulphate, containing 27% of magnesium oxide, 6% of alumina, 61% of silica, and 4% of calcium, together with water and other impurities, is formed, and fired at 1200—1230°. T. S. Wheeler.

Production of cemented acid- and alkali-resisting vessels. B. Ehrig & Co. (G.P. 438,879, 26.10.24).—The inner walls of the vessels are coated with an acid- and alkali-resisting surface formed by heating refractory material to which a suitable flux or glazing powder has been added. L. A. Coles.

Kilns for burning clay products, glazed ware, and pottery. H. Webster (E.P. 269,344, 17.10.25).—A battery of kilns of the continuous type is provided with central longitudinal flues connecting adjacent kilns, a parallel flue passing alongside all the kilns of each series, and a pair of transverse flues between adjacent kilns in operative communication by means of dampers with the longitudinal and parallel flues and with a chimney. B. W. Clarke.

Apparatus and process for burning ceramics and the like. H. Koppers, Assr. to Koppers Development Corp. (U.S.P. 1,631,536, 7.6.27. Appl., 31.8.21. Conv., 21.8.19).—See G.P. 349,951 ; B., 1922, 711 a.

IX.—BUILDING MATERIALS.

Re-use of plaster of Paris moulds. M. Farnsworth (Ind. Eng. Chem., 1927, **19**, 714—717).—Plaster of Paris moulds used for die-casting metals become pitted and dehydrated by the heat and unfit for further use. The anhydrite formed can be rehydrated to gypsum and recalcined (cf. B., 1925, 804), but the regenerated moulds show a large decrease in tensile strength. Changes in the character of plaster during five recalcinations were studied by determinations of water of crystallisation, water-carrying capacity, setting time, and tensile strength, but the cause of loss of tensile strength does not appear to be a chemical one. Examination of the regenerated samples of calcined plaster and gypsum by an *X*-ray photographic method showed no changes in the particle size of the former, but the crystals in the gypsum became continuously larger with each successive calcination, due, it is believed, to incomplete calcination, the particles of uncalcined gypsum acting as centres of crystallisation. This effect may to some extent be obviated by adding to the ground plaster, before recalcination, a small amount of aluminium oxide, the optimum proportion being about ¼%, but this or further additions of the oxide do not prevent further loss of tensile strength upon second recalcination. C. O. Harvey.

Concrete deterioration, more especially with reference to its use for conduits and in lining iron pipes. J. R. Baylis (Amer. Soc. for Municipal Improvements, 1926, 217—234).—The extent of the carbonation of the calcium compounds in set cement or concrete exposed to the air or to water is an important feature in determining the resistance of the concrete to deterioration. Not only is the calcium carbonate formed less resistant to the attack of saline waters than the original high calcium silicates, but its formation results in setting free the sulphates in the concrete with the result that needles of calcium sulphoaluminate are formed which rapidly disintegrate the content. The original SO_3 content of the cement or water is not a sufficient indication of the durability of the concrete, since the distribution of the pores may result in local concentrations of sulphates with consequent disintegration. In view of the possibilities of deterioration it is pointed out that for conduits especial care must be taken to make good dense concrete ; for the concrete lining of cast-iron water-pipes, the usual thickness of about $\frac{1}{16}$ in. is not sufficient, except for non-corrosive waters, and a thickness of ½—1 in. concrete would be a more suitable lining. B. W. Clarke.

Patents.

Production of cements. I. G. Farbenind. A.-G. (E.P. 263,124, 6.12.26. Conv., 15.12.25).—The slag obtained by fusing a crude phosphate with sand and carbon is tapped off and mixed with calcareous or aluminous materials in proportions suitable to form a cement clinker. B. W. Clarke.

Production of porous concrete. E. I. Lindman (E.P. 262,394, 11.10.26. Conv., 5.12.25).—Clay or clayey material is calcined at above the sintering temperature and the operation interrupted before the expanded mass begins to shrink ; on cooling, the clinker produced is ground and used as an admixture with cement, effervescing means, and water in the production of porous concrete. Preferably, the mixture is calcined at such temperature that the mass is melted, a further supply of gases being developed which remain occluded in the material. The light product obtained on cooling (*d* 0·5) has 100% increased heat-insulating properties compared with porous concrete produced in the usual way. B. W. Clarke.

Production of a hydraulic aluminous binding material and an iron alloy. L. de Lambert, Assr. to P. Zucco (U.S.P. 1,628,872, 17.5.27. Appl., 17.4.24).—Bauxite (47 pts.), limestone (45 pts.), phosphorite (8 pts.), and carbon (10—15 pts.), heated at 800°, yield impure iron and a hydraulic cement. T. S. Wheeler.

Process and apparatus for tempering plaster. L. E. Chassevent (E.P. 266,335, 11.2.27. Conv., 20.2.26).—Plaster is mixed with hot water at 60—100°. The plaster can be kept for several hours in this condition without affecting its setting properties, and when used it will harden rapidly when the temperature falls to 38—43°. B. W. Clarke.

Continuous [brick] kiln and drier. C. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. (U.S.P. 1,627,841, 10.5.27. Appl., 21.2.20).—Bricks are burnt in a fire-heated tunnel kiln, the products of combustion being utilised in drying the bricks before

burning. The bricks and transporting device are air-cooled, the heated air being injected into the drying chamber at intervals along its length in order to supply heat thereto and to serve as a vehicle for carrying away moisture from the bricks. B. W. Clarke.

Preservation of stone, brick, and like work. W. Anderson (E.P. 271,203, 17.3.26).—A mixture of silicates and silicofluorides with or without the addition of fluorides is claimed. B. W. Clarke.

Wood preservative. L. P. Curtin, Assr. to Western Union Telegraph Co. (U.S.P. 1,624,930, 19.4.27. Appl., 28.12.25).—Barium hydroxide solution emulsified with petroleum is employed to impregnate wood. T. S. Wheeler.

Preservative for telegraph poles etc. J. Himmelsbach (G.P. 435,146, 28.9.24).—An addition of 10% of natural bitumen is made to the mixture of coal-tar pitch and petroleum pitch used for the preservation of the wood of telegraph poles etc., the petroleum pitch being preferably one of very high m.p. A. B. Manning.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Production of iron sponge. M. Wiberg (Amer. Electrochem. Soc., April, 1927, **51**. Advance copy. 26 pp.).—The reduction of magnetite by carbon monoxide may be regarded as occurring in two stages (1) $Fe_3O_4 \rightarrow FeO$; (2) $FeO \rightarrow Fe$. With ore fed into the top of the furnace shaft and carbon monoxide introduced at the bottom, reaction (2) will occur in the lower part of the shaft and at 1000°, if equilibrium were attained, the gas leaving this zone would contain 29% CO_2. If this gas then produced the corresponding quantity of ferrous oxide by reaction (1) in the upper zone of the furnace, its carbon dioxide content would only reach 38%, whereas the equilibrium concentration for this reaction at 1000° is 82%. In the Wiberg process (cf. U.S.P. 1,401,222, B., 1922, 108 A) utilisation of the reducing capacity of carbon monoxide is attained by removing a large proportion of the gas leaving the lower zone, enriching it in carbon monoxide by passing through hot coke or charcoal (water may also be added to produce some hydrogen), and then returning it to the bottom of the shaft. Low-grade fuels may be used for this purpose, any sulphur derived from the coke being removed by passing the gases through a layer of lime. By removing 78% of the gas in this way, the remainder, supposing it to contain 29% CO_2, would, if used completely in reducing the corresponding amount of magnetite, leave the furnace containing 72% CO_2. Actually gases containing about 20% CO_2 were withdrawn and the carbon dioxide content of the remainder rose to about 50% in the upper zone. The carbon monoxide not utilised in reduction is burnt by introducing an air blast above the upper reduction zone, whereby the incoming ore is preheated to an extent which is regulated by the composition of the gas. Semi-commercial trials were carried out at Sandviken, Sweden. The regenerator was electrically heated and reduced the carbon dioxide content of the gas to 1% or less, its temperature being maintained above 1000°. The iron sponge is obtained as lumps of about the same size as those of the ore, and may contain over 1% C. On smelting, the carbon monoxide produced by the oxidation of this carbon protects the iron against oxidation. If the carbon content of the sponge is high enough, good quality steel may be produced by smelting in the crucible or electric furnace without addition of pig iron. Steel produced from iron sponge is of a higher quality than that obtained from ordinary raw materials, probably because the sponge has never been in a molten condition during its production, and therefore has not been able to form slag inclusions. Foreign material in the ore remains as relatively big grains in the sponge, and easily rises to the surface on smelting. The sponge obtained from low-grade ores (30—50% Fe) may be treated by magnetic separation to remove the gangue (which contains most of the sulphur and phosphorus). The powdered iron sponge is readily compressed into solid briquettes without addition of a binder. For the production of 1000 kg. of iron sponge from ore containing 95·3% of magnetite using coke in the electric regenerator, 222 kg. of coke was used and 1280 kw.-hrs. of electrical energy, the electrode consumption being only 2 kg. In places where electrical energy is expensive the electric regenerator could be replaced by two "alternating" producer-gas generators. H. J. T. Ellingham.

[Production of steel by] carbonising scrap iron. R. Hennecke (Stahl u. Eisen, 1927, **47**, 777—780).—Good quality steel may be produced by melting scrap iron and steel without addition of cast iron scrap or raw pig iron. The scrap is briquetted together with 1—2% of carbonaceous material, as free as possible from sulphur, the briquettes being melted with 3% of limestone in an acid-lined open-hearth furnace. When the charge has melted down and the evolution of gas has almost ceased 1% of fluorspar and a further 3% of limestone are added in two portions to assist in the removal of sulphur. Finally the steel is deoxidised in the ladle by the addition of 5—7 kg. of 80% ferromanganese per ton of steel. The output of steel per hour is about 5·8—5·9 tons per 20 $m.^2$ of hearth. Cost data compare favourably with those of the usual process. A. R. Powell.

Action of pure carbon monoxide on iron at elevated temperatures. W. P. Fishel and J. F. Wooddell (Trans. Amer. Soc. Steel Treat., 1927, **11**, 730—740).—On heating samples of Armco iron in carbon monoxide at temperatures from 800° to 1100° the amount of carbon dioxide formed was greater than that given for the equilibrium values for the system CO–CO_2–C. Iron carbide was formed during the reaction, which was reversible. High temperature gave a low-carbon case with deep penetration, low temperature produced a high-carbon case with low penetration. The rate of penetration of carbon monoxide and dioxide into an Armco cylinder with $\frac{3}{32}$ in. walls was measured at 950—1100°. The rate for both gases increased with rising temperature, but more quickly for the former. It would not, however, account for the depth of case produced by carbon monoxide. T. H. Burnham.

Properties of materials at high temperatures. I. Mechanical properties of Armco iron, 0·17% carbon steel, and 0·24% carbon steel, with special

reference to creep. H. J. TAPSELL and W. J. CLENSHAW (Dept. Sci. Ind. Res., Eng. Res., Spec. Rep. No. 1, 1927, 60 pp.).—Tensile, torsion, impact, hardness, and fatigue tests have been carried out on Armco iron, and on 0·17% and 0·24% carbon steels at temperatures up to 700°, and the phenomenon of creep has been specially investigated. In the tensile tests at normal rates of loading the ultimate strength reached a maximum between 200° and 250°. No yield point, as determined by the drop of the testing-machine beam, was evident above 250° for Armco iron, or above 200° for the carbon steels. Under a steady load the extension–time curves show different forms according to the stress and temperature. With a given load above a certain temperature the velocity of creep first decreases and then increases, the extension–time curves passing through a point of inflexion, but below a definite temperature the creep gradually ceases with time. In the same way at a given temperature a limiting stress can be found below which the creep ceases after a time, although this same stress produces a certain amount of initial creep. These limiting stresses are very sensitive to temperature; thus a change of 20° may cause a change of 20% in the limiting stress for Armco iron. There is considerable similarity between the torsion test and tensile test results, but the maximum value of the modulus of rupture occurs at a higher temperature than the corresponding maximum ultimate tensile strength. The effect of different types of notches in the impact test specimens was investigated. The fatigue tests, in a Haigh alternating-stress testing machine at 2400 cycles/min., showed that the fatigue ranges for zero mean stress, based on 10^7 reversals, are considerably higher at 300—400° than at air temperature, although the limiting creep stresses are falling rapidly at these temperatures. This is probably due to the rapid rate of reversal.

W. HUME-ROTHERY.

Graphite in grey cast iron. P. BARDENHEUER (Stahl u. Eisen, 1927, **47**, 857—867).—In grey cast iron of normal composition the volume of the precipitated graphite is relatively so great that its form and degree of fineness have a far more important effect on the mechanical properties than has the composition or structure of the ground mass. The tensile strength, bending strength, and resistance to shock are greater the finer and more evenly distributed are the graphite particles; coarse and irregular graphite inclusions not only impair the physical properties, but also reduce the resistance of the metal to atmospheric corrosion. The formation of coarse graphite lamellæ is hindered by the presence of the maximum amount of solid solution at the temperature at which the eutectic freezes, owing to the action of the solid solution in accelerating the rate of solidification of the eutectic. The absence of undissolved graphite nuclei is important if a fine structure is to be obtained; this is assured by keeping the carbon content relatively low or by heating the metal at a sufficient temperature to dissolve the carbon completely. Addition of manganese above 1%, chromium, and more especially sulphur, increases the stability of the carbide; aluminium, nickel, and silicon, on the other hand, decrease it. The simplest method of obtaining a high-grade cast iron, however, is to add sufficient steel scrap to the melt to reduce the carbon content to 1% below that of the eutectic composition.

A. R. POWELL.

Determination of silicon in steel and pig iron. A. STADELER (Stahl u. Eisen, 1927, **47**, 966—969).—Comparative tests of six methods used in ironworks for the determination of silicon showed that there is little to choose between the methods as regards accuracy provided that, in all cases, the filtrate from the first silica residue is evaporated again to recover traces that escape the first filtration. The most rapid and economical method is that involving evaporation with hydrochloric acid followed by evaporation of the weighed residue with hydrofluoric acid; in the case of pig iron, addition of bromine to the acid in which the metal is dissolved is recommended. Fusion of the silicious residue from the acid evaporation is unnecessary in any case, and serves only to increase possible sources of error.

A. R. POWELL.

Determination of molybdenum in iron and steel. E. FÄRBER (Chem.-Ztg., 1927, **51**, 171).—The sample (1—2 g.) of the metal chips is dissolved in hydrochloric acid (*d* 1·19), with subsequent addition of 2 g. of ammonium persulphate. The filtrate is boiled, saturated for 20 min. with hydrogen sulphide, and the molybdenum sulphide collected, washed with hot dilute hydrochloric acid and with hot water, dried, ignited, and weighed. The impure oxide so obtained is extracted for 1 hr. with 15% potassium hydroxide solution, and the insoluble oxides of copper and iron are collected, dried, ignited, and weighed, this weight being deducted from that of the impure oxides to give the weight of molybdenum trioxide.

F. R. ENNOS.

Formation of ferrite and diminution of impact resistance in tempered nickel-chromium steels. A. SCHLEICHER (Rev. Mét., 1927, **24**, 293—295).—After forging at 1100°, followed by annealing at 875°, quenching in oil, and tempering for 4 hrs. at 650°, the impact resistance of steels containing 0·45—0·51% C, 0·2—0·4% Cr, and 1·4—1·5% Ni, decreased from 8—10 kg./cm.^{2} to 0·9—1·2 kg./cm.^{2} This is ascribed to the formation of aggregates of acicular ferrite owing to the prolonged heating at a temperature near the Ac1 point. Subsequent annealing at 840° (A3 point) and cooling in air resulted in the formation of a Widmannstätten structure without improvement in the resistance to shock. A steel containing 0·26% C, 0·72% Cr, and 3·77% Ni, after a complicated forging and heat-treatment, followed by an annealing operation at 660° for 3 hrs., showed large columnar masses of ferrite in the microstructure and a correspondingly low impact resistance. Hence steels of the above composition should not be tempered, after forging, at a temperature too near the A1 point, and the period of heating must be as short as possible.

A. R. POWELL.

Effect of tin on the mechanical properties of copper. W. STAHL (Chem.-Ztg., 1927, **51**, 427).—Addition of a small quantity of tin, *e.g.*, 1%, to tough-pitch copper removes any adsorbed hydrogen and carbon monoxide and increases the sp. gr. from 8·625 to 8·913; at the same time the tensile strength is improved and the

ductility only slightly reduced. Overheating during annealing of this alloy, however, induces brittleness and internal fractures due to segregation of a constituent rich in tin. A. R. Powell.

Determination and separation of the oxidisable constituents of aluminium alloys. G. Jander and F. Baur (Z. angew. Chem., 1927, **40**, 488—490. Cf. Jander and Wendehorst; A., 1922, ii, 529).—When hydrogen chloride free from oxygen is passed over aluminium alloys containing magnesium at 200°, iron, copper, manganese, and part of the magnesium remain behind as metals or chlorides. Aluminium and silicon chlorides volatilise with some magnesium chloride which is carried over as a fine dust. By application of an external electric field to a dust-settling chamber through which the mixture passes, practically the whole of the magnesium chloride dust is deposited therein. The analysis of the various fractions is carried out by usual methods. When pure hydrogen chloride is used in this way to decompose commercial aluminium, the whole of the silicon is found as chloride, no silica remaining in the reaction boat. L. M. Clark.

Aluminium cans for foods. Serger.—See XIX.

Patents.

Manufacture of pure iron. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 269,677, 23.1.26).—Iron carbonyl in a liquid or vapour state is introduced into a vessel heated preferably at 250—300° in such manner that decomposition is effected mostly in the hot free space and not by contact with the walls of the vessel. Temperatures above 400° are to be avoided because of the interaction between carbon monoxide and iron at such temperatures. Gases or other substances which act catalytically may be introduced to assist the decomposition. C. A. King.

Production of dense iron and iron alloys directly out of oxide ores. H. G. E. Cornelius (E.P. 252,677, 26.3.26. Conv., 26.5.25).—Briquettes formed from a mixture of finely-divided iron ore, carbonaceous material, and vanadium or titanium oxide are reduced and melted while floating on a slag bath in an electric furnace. The source of vanadium may be slag produced in a previous heat. C. A. King.

Gaseous reduction of iron ores. I. G. Farbenind. A.-G. (F.P. 613,677, 30.3.26. Conv., 11.4. and 1.7.25). —The reducing gases used in the process are obtained by treating low-grade solid fuels in powder form or vapours, or gases containing hydrocarbons, with a deficiency of oxygen or gases rich in oxygen. The reduced iron may be melted by means of the same gases, and the issuing gases from the melting operation used for reducing further quantities of ore, if necessary, after the addition of steam or other gas. The final spent gases may be utilised in the synthesis of ammonia or of methyl alcohol. A. R. Powell.

Reduction of metal oxides, especially iron ores. A./S. Norsk Staal (Elektrisk-Gas-Reduktion) (G.P. 438,558, 13.10.25).—Iron ore is reduced by means of water-gas in a closed circuit from which a portion of the used gases is periodically removed and treated with steam at a high temperature, whereby the carbon monoxide present is oxidised to carbon dioxide with the liberation of hydrogen. After removal of the carbon dioxide, the remainder of the gas is passed back into the circuit with the other gases, which have meanwhile been regenerated in the usual way. The whole of the oxygen of the ore is thus removed as carbon dioxide and water. A. R. Powell.

Alloy. V. Fener (U.S.P. 1,626,925, 3.5.27. Appl., 14.1.26).—To an alloy of 100 pts. of copper and 3—25 pts. of tin, while in the molten state, are added 1 pt. of ammonium chloride and 0·02—0·10 pt. of potassium ferrocyanide. F. G. Crosse.

Wrought metal article. W. H. Bassett, jun., Assr. to American Brass Co. (U.S.P. 1,630,999, 31.5.27. Appl., 28.1.26).—An alloy is used consisting of 95·5% Cu, 2% Sn, 2·5% Al, approximately, the aluminium being in excess of the tin. F. G. Crosse.

Soldering aluminium. S. Smith (E.P. 270,453, 10.12.26).—A solder for aluminium consists of a mixture of tin and zinc, the zinc being in greater proportion than tin, but not exceeding 70%. Vaseline or petroleum jelly is used as a flux. C. A. King.

Aluminium alloys. W. Guertler and W. Sander, Assrs. to T. Goldschmidt A.-G. (U.S.P. 1,629,699, 24.5.27. Appl., 6.5.26. Conv., 22.11.23).—Aluminium is alloyed with 4—26% by wt. of a magnesium–zinc compound, $MgZn_2$, and subjected to annealing, quenching, and ageing. H. Royal-Dawson.

Light aluminium alloy. H. G. C. Fairweather. From Établ. Métallurgiques de la Gironde (E.P. 270,824, 17.2.26).—A light alloy of high tensile strength and high coefficient of expansion contains 1·5—5 (2)% Cu, 0·3—3 (0·37)% Ni, 0·2—2 (0·27)% W, 0·2—2 (0·45)% Mg, and 97·8—88 (96·91)% Al (commercial). C. A. King.

Alloys for use as platinum substitutes. H. Limbourg (E.P. 270,974, 27.11.26).—Alloys suitable for use in place of platinum comprise 75—85% Au, 10—15% Pd, and 2·5—10% Pt. The m.p. varies from 1300—1400° according to the composition, and the alloys resist the action of any single mineral acid and of fused alkali carbonates and nitrates, but are slightly attacked by fused alkali hydroxides. A. R. Powell.

Treatment of ores. J. T. Terry, Assr. to T. H. Sheridan (U.S.P. 1,627,582, 10.5.27. Appl., 7.9.22).—Ground ore containing silver is treated with an ammoniacal solution of copper sulphate and sodium thiosulphate, the extracted silver being recovered by treatment of the filtrate with sodium sulphide. T. S. Wheeler.

Method of metallising ores. H. N. Tracy (U.S.P. 1,628,012, 10.5.27. Appl., 29.5.23).—Ground ore is heated in a reducing atmosphere, and is introduced by a centrifugal thrower into the bottom of a bath of molten lead. The metal present alloys with the lead, and the gangue which rises to the surface is removed in a gas current. Means are provided for maintaining a reducing atmosphere in contact with the surface of the bath. T. S. Wheeler.

Manufacture of malleable nickel. N.V. Hybinette (U.S.P. 1,628,149, 10.5.27. Appl., 17.1.21).—In the production of malleable nickel by electrodeposition,

use is made of an anode of nickel containing 1% C, 1% Si, and 1% S, and of an aqueous electrolyte containing nickel chloride (40—60 g./litre), and boric acid (10—20 g./litre). A current density of 5—200 amp./sq. ft. is employed at a temperature of 40—50°. A portion of the electrolyte is continuously removed, neutralised by boiling in presence of nickel carbonate, filtered, and returned to the process. The deposited metal is rendered homogeneous by hot-working.

T. S. Wheeler.

Manufacture of nickel catalyst. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 255,884, 22.7.26. Conv., 22.7.25).—A highly active catalyst is formed by precipitating nickel, or a mixture of nickel and a metal of the iron or silver group, in the form of a crystalline hydroxide or carbonate in the presence of a carrier, *e.g.*, silica gel. The temperature for precipitation is preferred at about 70°. The product is reduced in hydrogen at about 400°.

C. A. King.

Production of molybdenum and tungsten trioxides. P. Schwarzkopf (U.S.P. 1,629,004, 17.5.27. Appl., 23.7.26. Conv., 26.4.26).—Ore containing molybdenum or tungsten compounds is heated in air to form the respective trioxide, which, when oxidation is complete, is sublimed into a condensing chamber in a current of air.

T. S. Wheeler.

Recovering antimony from alloys. L. and M. Meyer (Hüttenwerke Tempelhof A. Meyer) (E.P. 264,139, 9.12.26. Conv., 8.1.26).—A quantity of sulphur equivalent to all the metals except antimony is added to the alloy when in a molten condition, molten antimony being separated from the other metal sulphides.

C. A. King.

Reclaiming [easily oxidisable] metals. T. D. Stay and C. O. Tessier, Assrs. to Aluminum Co. of America (U.S.P. 1,630,361, 31.5.27. Appl., 28.10.21).—Light, readily oxidisable metals are melted in a furnace provided with mechanical agitation. A continuous feed of scrap metal is maintained to the furnace, and means for concentrating the slag on the surface of the molten metal are provided.

C. A. King.

Protection of molten baths of easily oxidisable metals. G. Michel (E.P. 257,221, 10.2.26. Conv., 18.8.25).—Molten baths of easily oxidisable metals, *e.g.*, magnesium, are protected by a covering of a suitable salt, *e.g.*, magnesium fluoride, which is sprinkled with sulphur to form a local non-oxidising atmosphere.

C. A. King.

Tough annealing of metal alloys. J. L. La Cour and F. O. M. Lindh (E.P. 243,006, 13.11.25. Conv., 13.11.24).—Non-ferrous alloys containing copper, zinc, tin, lead, and aluminium are heated at a suitable annealing temperature in a neutral atmosphere and then slowly cooled to a temperature below 75% of the annealing temperature, before the alloy is exposed to the atmosphere.

C. A. King.

Treatment of zinc-bearing ores for the recovery of zinc by electrolytic deposition. Electrolytic Zinc Co. of Australasia, Ltd. (E.P. 258,575, 8.9.26. Conv., 17.9.25).—Prior to the recovery of zinc by electrodeposition an impure solution of zinc is treated with zinc dust while passing through an enclosed vessel in which the solution is agitated, sufficient time being allowed for the precipitation of copper, cadmium, etc. without the production of basic zinc sulphate. Excess of zinc dust is separated by gravity, and the solution containing suspended impurities is filtered.

C. A. King.

Electric bright-annealing furnace. Siemens-Elektrowarme-Ges.m.b.H. (E.P. 262,468, 4.12.26. Conv., 5.12.25. Addn. to E.P. 152,405; B., 1927, 115).—In a furnace constructed as in the prior patent, the resistance heating winding is arranged within the protective bell.

J. S. G. Thomas.

Production of rustless iron and steel. D. W. Berlin, Assr. to Aktiebolaget Ferrolegeringar (U.S.P. 1,631,512, 7.6.27. Appl., 20.7.23. Conv., 28.8.22).—See E.P. 202,952; B., 1924, 717.

Aluminium[-copper-silicon] alloy. V. Fuss, Assr. to Ver. Aluminiumwerke A.-G. (U.S.P. 1,631,930, 7.6.27. Appl., 31.5.24. Conv., 16.8.23).—See E.P. 220,602; B., 1925, 886.

Electro-deposition of chromium and its alloys. Olausson & Co. Aktiebolag (E.P. 264,442, 17.3.26. Conv., 14.1.26).—See F.P. 612,880; B., 1927.

[Carriers for heating elements of] electric resistance annealing furnaces. A.-G. Brown, Boveri, & Cie. (E.P. 271,003, 13.1.27. Conv., 19.5.26).

Apparatus for sherardising metal articles. Rustproof Processes, Ltd., and M. Trouton (E.P. 269,680, 25.1.26).

Iron alloy (U.S.P. 1,628,872).—See IX.

XI.—ELECTROTECHNICS.

Action of iron as an impurity in the lead accumulator. I. Capacity loss due to self-discharge. II. Permanent capacity loss. Adsorption and desorption of the iron by the positive plate. F. M. Lea and J. T. Crennell (Trans. Faraday Soc., May, 1927. Advance proof).—I. The presence of iron in the electrolyte of a lead accumulator causes (*a*) a loss of capacity as measured by discharging immediately after charging (permanent loss), and (*b*) a further decrease in capacity when allowed to remain for some time before discharging (self-discharge loss). No other effect of iron on the life of performance of the cells has been found. The self-discharge loss increases notably with temperature, and at given temperature is practically proportional to the iron concentration, but at the highest temperature used (48·9°) the effect of iron is masked by losses due to other causes. For a cell with pasted plates and a capacity about 17 amp.-hrs. at 23·9°, an iron concentration of 0·84 g./litre caused an average self-discharge loss during 9 days of 0·22 amp.-hrs./day, the rate of self-discharge decreasing slightly during this period. There is evidence that self-discharge occurs to the same extent at both positive and negative plates. There is a definite tendency for iron to accumulate in the lower layers of the electrolyte, this being attributed to the action of concentration cells caused by the higher acid concentration at the bottom of a recently-charged cell. Assuming that

self-discharge is due to alternate oxidation and reduction of iron at the respective plates (Dolezalek, "The theory of the lead accumulator," 1904), an approximate relation is deducted between the rate of self-discharge and the cell dimensions, iron concentration, and the diffusion coefficients of ferrous and ferric ions; this gives self-discharge rates of the order observed. The view that iron also acts by accelerating the reaction $Pb + H_2SO_4 = PbSO_4 + H_2$ at the negative plate is not supported. II. For a cell of 17 amp.-hr. capacity at 23·9°, the permanent loss caused by 0·52 g. of iron per litre was 5% of the total cell capacity when discharging at a 5 hr. rate. This permanent loss is practically proportional to the iron concentration, and increases with increase of temperature or decrease of rate of discharge. It is connected with the adsorption of iron by the positive plate. Iron is readily adsorbed by lead peroxide, especially if freshly precipitated, and the adsorption increases with temperature, but decreases with increasing acidity of the solution. The adsorption is largely irreversible, but desorption occurs during the electrical working of cells in which the iron in the electrolyte and the paste are not in equilibrium. Removal of iron from the system by successive changes of electrolyte removes the permanent loss. The probable mechanism of the production of the permanent loss is discussed. The titanous chloride method of determining iron has been adapted to the determination of small amounts of iron in sulphuric acid solutions.

H. J. T. Ellingham.

Electric furnace for gas reactions. O. Scarpa (Giorn. Chim. Ind. Appl., 1927, **9**, 167—168).—In the usual types of electric furnaces for gas reactions, replacement of the metallic electrodes by carbons renders possible the lowering of the voltage of the arc, but the necessity of either injecting violent gas currents to dilate the arc sufficiently or employing complicated and costly devices remains. This necessity may be avoided by arranging the one carbon electrode perpendicular to the plane of three or four electrodes of the opposite sign, so that three or four arcs are formed. The voltage used is of the order 80, the intense electrodynamic actions induced by the arrangement causing great elongation of the arcs in the direction of the single electrode.

T. H. Pope.

Platinum-wound resistance furnace. E. Orton, jun., and J. F. Krehbiel (J. Amer. Ceram. Soc., 1927, **10**, 373—387).—A description is given of an endeavour to construct a furnace with an effective hot chamber 4—5 in. long and 3 in. in diameter, in which the temperature would be uniform throughout, the control accurate, the atmosphere clean and oxidising, and able to melt cone 20. The furnace has not been working long enough to furnish knowledge of its practical value.

A. T. Green.

Flames of atomic hydrogen. I. Langmuir (Ind. Eng. Chem., 1927, **19**, 667—674; cf. B., 1926, 549, 550).—The maximum temperature of the atomic hydrogen flame is calculated to be 4030° Abs., the degree of dissociation at this temperature being 0·642. When acetylene burns with an equal volume of oxygen the flame may attain 3270° Abs., the flame gas containing (by vol.) 60·9% CO, 21·8% H (molecular), and 17·3% H (atomic). The rate at which heat is delivered to a tungsten or copper surface by the atomic hydrogen flame produced by a 60 amp. arc (1330 watts/cm.^{2}) is 26 times as great as that from a Bunsen burner flame, and about double that from the oxy-acetylene flame. The behaviour of refractory substances in the flame is described. Except in the case of difficultly volatile metals (*e.g.*, molybdenum and tungsten), cooling by evaporation limits the temperature to which molten metals can be heated by allowing the atomic-hydrogen flame to play on the metal surface; the following maximum temperatures were observed (° Abs.): nickel, 2870° (b.p. 3650°); copper, 2840° (b.p. 3110°); silver, 2660° (b.p. 2740°).

S. K. Tweedy.

Patents.

Preparation of an electron-emitting cathode. F. Holborn, Assr. to Hazeltine Corp. (U.S.P. 1,625,776, 19.4.27. Appl., 14.1.26).—Natural guaiac is mixed with barium or strontium hydroxide or carbonate and water, and the mixture is applied to a thermionic valve filament, which is then heated first at 200° and then at 1100°.

T. S. Wheeler.

Metal-coated [arc] electrode. E. L. Swift, Assr. to V. Swift (U.S.P. 1,626,104, 26.4.27. Appl., 21.2.22).—Carbon mixed with an organic binder, a mineral acid, and a halogen salt is formed into an arc electrode, and is plated first with copper and then with a thin layer of brass, nickel, silver, or the like, to protect the copper from oxidation. T. S. Wheeler.

***X*-Ray filter.** A. St. John, Assr. to Union Carbide & Carbon Research Laboratories, Inc. (U.S.P. 1,624,443, 12.4.27. Appl., 20.10.21).—An *X*-ray filter for use with a molybdenum target comprises a screen coated with calcium zirconium fluoride, which has been rendered fluorescent by strong ignition.

T. S. Wheeler.

Neon tube. R. R. Machlett, Assr. to Rainbow Light, Inc. (U.S.P. 1,628,256, 10.5.27. Appl., 16.10.26).—The life of a neon tube is increased by forming the electrodes with cavities containing magnesium carbonate, so that during operation there is a slow evolution of carbon monoxide. T. S. Wheeler.

Metal filaments. T. E. Foulke, Assr. to General Electric Co. (U.S.P. 1,628,456, 10.5.27. Appl., 29.7.22).—Tungsten wire is passed through a furnace at 950°, so that a film of oxide is formed on it, and is then coated with a getter. T. S. Wheeler.

Activation [of thermionic valve filaments] by means of hydrogen-free, carbon-bearing gas. J. W. Marden, T. P. Thomas, and J. E. Conley, Assrs. to Westinghouse Lamp Co. (U.S.P. 1,626,685, 3.5.27. Appl., 10.4.23).—Thoriated tungsten wire for thermionic valve filaments is activated by passage at a dull-red heat through carbon monoxide or cyanogen.

T. S. Wheeler.

[Filling for] gas-filled electric lamps. Siemens & English Electric Lamp Co., Ltd., and P. D. Oakley (E.P. 270,900, 10.5.26).—The harmful action of traces of water vapour in gas-filled lamps in which a tungsten filament is raised to incandescence in an atmosphere of nitrogen or argon or a mixture of these gases, is overcome by admitting into the lamp a small proportion,

e.g., 0·5—1%, of an oxyhalide of carbon, *e.g.*, carbonyl chloride, which may be introduced directly into the lamp or produced synthetically therein by the interaction of carbon monoxide and chlorine. J. S. G. THOMAS.

Depolariser for primary batteries. G. W. ARMSTRONG, Assr. to NATIONAL CARBON CO., INC. (U.S.P. 1,624,460, 12.4.27. Appl., 28.2.25).—Copper oxide is treated at below 100° with hydrogen sulphide until a sulphur content of 0·5% is reached. The product is of value as a depolariser for primary cells with an alkaline electrolyte. T. S. WHEELER.

Electrolytic cell. A. K. CROAD. From JESSUP & MOORE PAPER CO. (E.P. 270,104, 2.6.26).—An electrolytic cell, designed more especially for the production of caustic soda, comprises a casing supporting outer and inner cathode members having the form of a perforated iron basket. The inside of the outer member and the outside of the inner member are lined with asbestos or other material serving as a diaphragm which, when assembled, forms an asbestos-lined endless channel into which anode slabs of graphite are inserted, these being spaced so as to allow thorough circulation of electrolyte. A gas chamber or head adapted to clamp the cathode and diaphragm gas-tight between itself and the cell casing is provided. An additional outlet is provided for gaseous products liberated between the diaphragm and the outer casing, and another for liquid products at the bottom of the casing. Means comprising a float container automatically feed electrolyte, thus maintaining a constant level of electrolyte in the cell. J. S. G. THOMAS.

Electrolytic rectifier. L. J. KEELER, Assr. to LE R. P. BENSING and G. P. KOELLIKER (U.S.P. 1,628,785, 17.5.27. Appl., 18.1.26).—A permanent electrolytic rectifier comprises electrodes of nickel and magnesium, and an aqueous electrolyte containing 30% of potassium fluoride and 10% of sodium borate. T. S. WHEELER.

Apparatus for the electrical purification of gases. L. DRAULT and C. RAULOT-LAPOINTE (F.P. 614,442, 12.4.26).—The apparatus comprises a moving precipitation electrode kept clean by scrapers, a high-tension electrode directed towards the precipitation electrode, and means for cleaning the insulators. L. A. COLES.

Electrical gas-purifying plant, containing alternate discharge and precipitation electrodes placed transversely to the gas stream. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of C. HAHN (G.P. 438,834, 28.7.22).—The precipitation electrodes, all of which have the same mesh, are arranged in pairs, and between each pair is a sieve or set of sieves, the mesh of which decreases in the direction of the gas stream. L. A. COLES.

Mounting for electrodes of closed electric furnaces. I. G. FARBENIND. A.-G. (E.P. 263,756, 5.11.26. Conv., 28.12.25).

Insulating paper (U.S.P. 1,600,424 and E.P. 251,970).—See V.

Annealing furnace (E.P. 262,468).—See X.

Rubber [in cables] (E.P. 269,124).—See XIV.

Rubber deposits (E.P. 257,885).—See XIV.

Oxidation of compounds (E.P. 265,672).—See XX.

Silver from photographic solutions (U.S.P. 1,629,212).—See XXI.

XII.—FATS; OILS; WAXES.

Utilisation of waste in the fat-hardening industry. K. BUTKOVSKI (J. Oil and Fat Ind., Moscow, 1926, 11—12; Chem. Zentr., 1927, I, 821—822).—After removal of the nickel from the deposit of catalyst and adhering fat obtained in fat-hardening, the hitherto wasted residue, which amounts to 4% by wt. of the hardened fat and contains considerable amounts of kieselguhr from which the fat cannot be recovered by melting, is treated with hot lye (*d* 1·05), 70% of the fat being recovered as soap. S. S. WOOLF.

Change in sp. gr. of curd soap during the drying-out process. W. KRISTEN (Seifensieder-Ztg., 1926, **53**, 669—670, 689; Chem. Zentr., 1926, II, 3123).—The sp. gr. of soap curd (62—63%) rose, with shrinkage on drying out, from an initial value of 0·905 to 1·0482 in 39 days, and to 1·075 in 68 weeks. The outer layers were less dense than the mass in general. S. S. WOOLF.

Oxidation of linseed oil. II. G. F. HOLDEN (J. Soc. Dyers and Col., 1927, **43**, 157—158. Cf. B., 1918, 429 A).—Linoxyn films prepared by exposing linseed oil to atmospheric oxidation on roughened glass plates (*a*) at 18° for 12 days, and (*b*) at 100° for 8 hrs., were washed with ether, alcohol, and water, dried over concentrated sulphuric acid *in vacuo* to constant weight, and analysed. The figures for linoxyn obtained at 18° agree with the results of earlier observers, whilst that produced at 100° corresponds to a higher state of oxidation, and is more serviceable in industrial practice. Linoxyn has analytical figures agreeing closely with hexahydroxylinoleic triglyceride. S. S. WOOLF.

Gossypol in cottonseeds. GALLUP.—See XIX.

PATENTS.

Edible fat composition. H. E. DUBIN, Assr. to H. A. METZ LABORATORIES, INC. (U.S.P. 1,624,164, 12.4.27. Appl., 19.2.25).—The product, with m.p. below 37°, free from objectionable odour and taste, and of therapeutic value, comprises glyceryl margarate mixed with at least an equal weight of ethyl margarate. T. S. WHEELER.

Machine for preparation of artificial edible fats. G. SCHEFFLER (G.P. 437,670, 4.9.23).—Melting kettle, emulsifier, and cooling and kneading rollers are combined in one machine, in which, *e.g.*, the complete manufacture of margarine can be carried out. S. S. WOOLF.

Recovery of fats by extraction with volatile solvents, from emulsions stabilised with solid material. A. MARX (F.P. 609,806, 30.4.25).—The emulsion and the solvent circulate through apparatus comprising a suitable number of mixers and centrifugal separators for separating the fat from the solvent. L. A. COLES.

Apparatus for treating fish meal, guano, or the like products by means of a solvent for the recovery of residual oils or fats. G. W. BRADSHAW (E.P. 270,869, 30.3.26).—Material to be extracted is placed in a perforated cylinder, which is disposed in an outer cylinder so that an annulus is left between them. Solvent, under increased pressure or temperature if desired, is led into the material by means of a perforated central

tube placed axially in the inner cylinder, and percolates radially through the mass, the extract being discharged from the annulus. A filtering medium may be wrapped round the inner cylinder. The reverse process (of inward radial percolation) is also claimed.

S. S. Woolf.

Cooling apparatus for fatty emulsions and the like. W. G. Schröder (E.P. 269,416, 15.10.26).—Fatty emulsions are air-cooled by a reverse current of cold air in the jacket of a revolving drum which is nearly filled by an inserted hollow body, the fluid circulating through the annular space thus left, in a tortuous path induced by a series of staggered circumferential ribs on the inside walls of the drum. The external cooling means is improved by the provision of a heat exchanger, which is built up with the main cooler, and through which the cooling air is circulated. The receiver for the charge is fitted with a fluted roller which acts as stirring gear, producing a constant axial flow of emulsion. Solidified material is removed from the drum by means of a knife to which is given a reciprocating movement parallel to the axis of the drum, thus causing complete removal of the deposit and at the same time keeping the knife edge sharp. The apparatus may be used in the manufacture of margarine. S. S. Woolf.

Preparation of fats of a plastic and pliable consistency. K. Erslev (E.P. 269,384, 17.8.26).—Small proportions of gummy plasticisers, *e.g.*, unvulcanised rubber, rubber latex, chicle, etc., either alone or dissolved in suitable solvents which may be subsequently recovered, are incorporated into naturally occurring hard and brittle fats, *e.g.*, cacao butter, palm-kernel fat, rendering them pliable without greatly lowering their m.p. The process may be accelerated by leading a heated and preferably inert gas through the mass. S. S. Woolf.

Condensation apparatus for preparation of pale fatty acids. Continentale A.-G. für Chemie, Assees. of R. Tern (G.P. 436,890, 2.3.26).—A system of three connected rows of vertical air-coolers, those in the central row having greater cross-section than the others. By means of an air pump the volatile distillate is led to the top of the middle coolers, and the condensate is drawn off from the bottom of the outer ones.

S. S. Woolf.

Polymerisation of oils. S. Cabot (E.P. 250,538, 10.2.26. Conv., 10.4.25).—A readily polymerisable oil is heated with a non-volatile and more slowly polymerising oil in which it is soluble, the polymerised product of the first oil remaining colloidally dispersed in the second without gelatinising the mass. A typical example is the heating of 1 pt. by wt. of tung oil with 2 pts. by wt. of linseed oil for 1 hr. at 300°. S. S. Woolf.

Vitamins from cod-liver oil (U.S.P. 1,629,074).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Settling of oil paints. A. V. Blom (Farben-Ztg., 1927, **32**, 2082—2083).—Paste red lead was shaken up with linseed oil, alone and thinned with light petroleum, turpentine, and ether, the volume ratio pigment : medium being kept constant. As anticipated, the rate of settling varied inversely as the viscosity of the medium, whilst the final volume of sediment lay between the volume occupied by the packed dry pigment and the actual volume of solid particles. On pouring off the supernatant liquor, replacing it by various solvents, and re-shaking, the rate of settling and final volume of sediment were anomalous. The probable causes for these anomalies are discussed, the wetting, solvating, and flocculating action of the medium being considered.

S. S. Woolf.

Linseed oil stand oil. H. Wolff (Farben-Ztg., 1927, **32**, 2019—2020).—From consideration of the analytical constants of five samples of stand oil, the empirical relationships $2 \cdot 7\ R - D - 0 \cdot 143\ I =$ (approx.) 3045, and $I_o = (R - 1458 \cdot 5 + 0 \cdot 11\ I)/0 \cdot 224$ (where $R = 1000\ n_D^{20}$, $D = 1000\ d^{20}$, and $I =$ iodine value of the stand oil, whilst $I_o =$ iodine value of the original oil) are developed from earlier relationships of similar character due to Lund, saponif. value, which appeared in the latter's expressions, being incorporated into the constant term. By the use of these relationships, information may be obtained as to the purity of linseed oil stand oils in the absence, which must be established, of tung oil. S. S. Woolf.

Rubber pigments. Ditmar.—See XIV.

Patents.

Manufacture of a substitute for turpentine. L. L. Odom, Assr. to M-O-R Products Co. (U.S.P. 1,625,622, 19.4.27. Appl., 18.3.24).—A mixture of rosin (100 pts.) and calcium oxide (10 pts.) is heated at 190° for 2 hrs., petroleum distillate, d 1·53, is added at 80°, and the product distilled. T. S. Wheeler.

Quenching lithopone. A. S. Krebs, Assr. to Krebs Pigment and Chemical Co. (U.S.P. 1,630,267, 31.5.27. Appl., 29.6.23).—A continuous fine stream of hot lithopone is fed into a running stream of water which conveys the quenched pigment to a settling tank.

S. S. Woolf.

Drying and calcining lithopone. W. G. Graves, Assr. to Grasselli Chemical Co. (U.S.P. 1,630,613, 31.5.27. Appl., 23.1.26).—The entire drying and calcination of lithopone is carried out as a continuous operation in one vessel. S. S. Woolf.

Manufacture of a zirconium oxide-silica composite. C. J. Kinzie, Assr. to Titanium Alloy Manuf. Co. (U.S.P. 1,618,288, 22.2.27. Appl., 6.11.25).—A zirconium ore or mineral, *e.g.*, zirconium silicate, is heated with an equal weight of sodium carbonate at 900°, and the product is ground, washed with water, and treated with sodium chloride and a 50% solution of sulphuric acid. On being heated and stirred the mixture rapidly solidifies. It is calcined at 1000° and the product is ground, washed and dried. It consists of an intimate mixture of zirconium dioxide and silica, and is of value in the manufacture of pigments. T. S. Wheeler.

Writing fluids. Marconi's Wireless Telegraph Co., Ltd., Assees. of R. H. Ranger (E.P. 257,954, 4.9.26. Conv., 5.9.25).—A freely flowing, non-clogging, quick-drying writing fluid, particularly designed for use in radio-transmission of pictures, consists of a base of waxy material, *e.g.*, paraffin wax, to which is added colouring matter, *e.g.*, aniline dyes. The mixtures are usually

solid at ordinary temperatures, and must be heated above their m.p. before use. S. S. WOOLF.

Production and utilisation of dichroic inks. C. BITTINGER (U.S.P. 1,629,250, 17.5.27. Appl., 3.1.25). —A pigment, *e.g.*, Paris Green, is ground in a vehicle to form a coloured ink, with which an organic dye of a different colour, *e.g.*, Methyl Violet, is then mixed. The pigment colour is predominant until the ink is subjected to mechanical pressure, when the dye colour is obtained. By varying the pressure in printing with this ink it is possible to produce one or other colour.
T. S. WHEELER.

Purification of condensation products produced from phenols and aldehydes. BAKELITE G.M.B.H. (E.P. 248,726, 17.2.26. Conv., 6.3.25. Addn. to E.P. 247,956; cf. G.P. 431,514; B., 1926, 889).—Phenol-aldehyde condensation products are treated, in the presence of water, with metallic oxides or hydroxides, other than the alkali hydroxides or compounds capable of furnishing such substances, in amount sufficient to neutralise free phenols without combining with the artificial resins. The condensation products are precipitated by water or by aqueous solutions of substances of hydrotropic action. Solvents for the condensation products may be added prior to the treatment with oxides etc., and filling substances may be present. The formation of resin salts, to which the process described in the prior patent was liable, is avoided, and greater economy is attained. S. S. WOOLF.

Manufacture of synthetic resins. COMMERCIAL SOLVENTS CORP., Assees. of E. R. LITTMANN, B. K. BROWN, and W. J. BANNISTER (E.P. 250,265, 1.4.26. Conv., 1.4.25).—The multivalent metal salts of alkyl hydrogen phthalates, formed from an alkali alkyl phthalate and a soluble salt of the metal, are friable insoluble resins. The following salts of *n*-butyl hydrogen phthalate are described: *Zinc*, m.p. 150°; *lead*, pale amber; *ferric*, ruby-red; *cupric*, bluish-green.
C. HOLLINS.

Manufacture of homogeneous glass-like condensation products of carbamide and formaldehyde. K. RIPPER, Assr. to F. POLLAK (U.S.P. 1,625,283, 19.4.27. Appl., 9.9.22. Conv., 31.5.21).—See E.P. 181,014; B., 1923, 988 A.

Condensation product. H. C. P. WEBER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,630,365, 31.5.27. Appl., 24.9.20).—See E.P. 169,451; B., 1922, 978.

Grinding mill (E.P. 270,465).—See I.

Photographic varnish (E.P. 270,387).—See XXI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Nature of matured rubber. II. G. BRUNI and T. G. LEVI (Giorn. Chim. Ind. Appl., 1927, 9, 161—164). —The aqueous fraction, obtained when the acetone extract of matured slab rubber is shaken with ether and water, contains (1) 10% of a mixture of an α-aminovaleric acid and two α-aminohexoic acids, one giving a readily soluble and the other a sparingly soluble copper salt; these acids, which have no influence on the vulcanisation, are obtained also by similar treatment of smoked sheet. (2) About 20% of tetra- and pentamethylenediamines, which greatly accelerate vulcanisation. (3) More than 30% of potassium phenylacetate, which, like the potassium salts of other fatty and fatty-aromatic acids, is an energetic accelerator of vulcanisation; the corresponding sodium salts are less active in this respect. Potassium salts of aromatic acids are weak accelerators, and the sodium salts are almost inactive. The ethereal fraction of the acetone extract contains phytosterol, which does not affect vulcanisation.
T. H. POPE.

Röntgen-spectrography of rubber and of similar extensible substances. Amorphous rings and their alterations with extension. J. R. KATZ (Chem.-Ztg., 1927, **51**, 381—385).—A review of the subject dealing with the significance of the "amorphous spectra" of liquids, the behaviour of polymerides and substances with several similar side-chains, the broad amorphous rings shown by various forms of rubber, the dividing of these rings when the rubber is stretched, and the view of the structural condition of rubber and similar extensible substances as intermediate between that of the amorphous and crystalline states. D. F. TWISS.

***Hevea* latex. VI. Proteins in serum from frozen latex.** R. O. BISHOP (Malayan Agric. J., 1927, **15**, 27—34).—Fresh latex was frozen for several days until on thawing it gave a clear serum; this showed the same p_H as the original latex, viz., 6·5. By successive acidification to p_H 4·5, saturation with sodium chloride, and heating at 100° three protein substances were obtained designated A, B, and C. After purification by dialysis, protein A retained a compound containing phosphorus and nitrogen which was extractible by alcohol. No indication of a lipin complex was obtained. The serum had d^{30} 1·015 (average), and the total yield of proteins was 0·6% by wt. of the serum.
D. F. TWISS.

Determination of sulphur in rubbers. E. KAHANE (Caoutchouc et Gutta-Percha, 1927, 24, 13,549—13,550). —1 g. of the sample is added to a mixture of 10 c.c. of nitric acid and 5 c.c. of perchloric acid. In the vigorous reaction, induced by gentle heating if necessary, the rubber disappears. The mixture is then heated strongly when it first darkens and then clears. After these operations, which occupy from 7 to 8 min., the precipitation of barium sulphate is effected in the customary manner. The use of a mixture of 5 c.c. of perchloric acid with 5 c.c. of sulphuric acid for the oxidation of 1 g. of rubber also enables a Kjeldahl determination of nitrogen to be completed in 10 min. D. F. TWISS.

Jelutong. B. J. EATON, C. D. V. GEORGI, and G. L. TEIK (Malayan Agric. J., 1927, **15**, 65—77).—Jelutong latex has p_H 7·0 (approx.) when fresh, but after 24 hrs. the value may attain 5·5. An average sample showed total solids 19·87%, ash 0·03%, coagulum (by alcohol) 15·54%, nitrogen 0·025%. The latex contains a large proportion of gummy material and sugars. Satisfactory coagulation of fresh latex can be effected by acidifying to p_H 5·0 with acetic acid (0·1 g. of acetic acid per 100 c.c. of latex), and boiling for a few minutes. Satisfactory coagulation is also produced by formic acid (0·05 g.), sulphuric acid (0·02 g.), and potash alum

(0·05 g.). The liability of jelutong to deterioration can be reduced by immersion in boiling water for 10 min. and then pressing. The removal of soluble organic matter thus effected reduces the tendency to fungoid development. D. F. Twiss.

Effect of the colour of inorganic and organic rubber pigments on organic accelerators of vulcanisation. R. Ditmar (Chem.-Ztg., 1927, **51**, 332—333).—The behaviour of a number of rubber mixtures, selected to reveal the influence of various organic accelerators on such pigments as vermilion, antimony sulphide, and a number of " Vulkan " organic colours, previous to or during vulcanisation between glass plates at 135°, indicates that this follows no simple general rule. It is necessary to make a practical test of the influence of each accelerator on each pigment under varying conditions. D. F. Twiss.

Barytes and its employment in the rubber industry. T. R. Dawson and N. H. Hartshorne (India-rubber J., 1927, **73**, 885—889, 926—930, 961—966).—Twenty samples of commercial barytes were submitted to a comprehensive chemical and physical examination. Particle size was investigated by elutriation and sedimentation methods, a simplified form of apparatus being described for the latter. Comparative vulcanisation experiments were made with two samples, the vulcanisates being examined as to tensile strength, elongation, stress–strain curve, permanent set, sp. gr., hardness, and colour. The results indicate that, for use in rubber, particle size is easily the most important feature, the practice of grading barytes according to colour bearing no rational relation to the properties desirable for rubber compounding. D. F. Twiss.

Patents.

Production of homogeneous rubber deposits from rubber latex. Anode Rubber Co., Ltd. (E.P. 257,885, 23.2.26. Conv., 4.9.25. Cf. E.P. 223,189; B., 1925, 46).—In order to avoid the anodic liberation of gases during the electro-deposition of rubber from rubber latex on a metallic anode such as zinc, cadmium, iron, or lead, the ammonia concentration of the latex should be at most 0·1*N*. Reduction of the proportion of ammonia in preserved latex can be effected by dialysis, aeration, partial evaporation, addition of formaldehyde (with formation of hexamethylenetetramine) or other carbonyl compounds, or even neutralisation. Alternatively, latex may be used which has been preserved with a less ionised substance, either alkaline or germicidal. Mere dilution of ammonia-preserved latex is not satisfactory as it adversely affects the deposition of the rubber. A further alternative is to effect deposition under such conditions that the anode potential is insufficient for electrolysis with gas formation; this, however, involves slow deposition. The condition of the rubber deposit formed can be modified by previously coating the anode with a liquid-absorbing substance, *e.g.*, gelatin or collodion; the anode surface also may be provided with any desired pattern, and individual parts may be differently constituted so as to give a porous or smooth rubber deposit. D. F. Twiss.

Manufacture of rubber goods. Anode Rubber Co., Assees. of P. Klein and A. Szegvári (E.P. 253,069, 12.1.26. Conv., 5.6.25).—In order to avoid the possibility of difficulty arising from undesirable features in certain ingredients for latex mixtures used in the manufacture of goods by electrophoretic methods, these ingredients are combined with a conditioning substance or substances such that in the particles of the resulting combination the undesirable characteristics are diminished. For example, oils or litharge may be blended with kieselguhr; sulphur may be used with kieselguhr, lamp-black, clay, or even with vulcanised rape oil; zinc oxide may be used as a dispersion of the product obtained by heating with sulphur and rape oil. [Reference is directed, in pursuance of Sect. 8, Sub-sect. 2, of the Patents and Designs Acts, 1907 and 1919, to E.P. 254,765.] D. F. Twiss.

Production of rubber goods directly from [rubber] latex. Anode Rubber Co., Ltd. (E.P. 252,673, 23.2.26. Conv., 30.5.25).—When a porous mould is immersed in rubber latex, preferably concentrated and, if desired, compounded, a layer of agglomerated rubber forms on the surface. The agglomeration may be increased by applying suitable agents such as acetic acid, calcium chloride, or zinc chloride to the side of the mould which is not in contact with the latex; it may also be aided by accelerating the migration of the serum through the mould, *e.g.*, by using a hollow mould and applying a slightly reduced pressure or promoting evaporation in the interior. Water-absorbing substances also may be introduced inside the porous mould. D. F. Twiss.

Rubber compounds and articles. Simplex Wire & Cable Co., Assees. of C. R. Boggs and J. T. Blake (E.P. 269,124, 24.7.26. Conv., 6.4.26).—Composite rubber products resistant to the action of ozone are made with an elastic portion imparting the desired physical characteristics covered with a protective plastic portion free from internal stress. A suitable resistant plastic gum comprises balata (35 pts.), reclaimed rubber (5 pts.), and oil substitute (60 pts.); the elastic inner material may be of any of the customary rubber " compounds." The invention is of especial value in connexion with insulated electrical cables. D. F. Twiss.

Manufacture of vulcanite and of composite materials, including vulcanite. H. P. Stevens (E.P. 269,693, 18.1.26).—Vulcanite is produced by heating a mixture of rubber, sulphur, and a powerful accelerator, *e.g.*, zinc diethyldithiocarbamate, at a low temperature, *e.g.*, below 100°, for a prolonged period, such as 10 days. The mixture may contain other additional ingredients, and may be converted into solution for coating articles before vulcanisation. D. F. Twiss.

[Precipitation] treatment of rubber. W. G. O'Brien, Assr. to Goodyear Tire & Rubber Co. (U.S.P. 1,628,326, 10.5.27. Appl., 22.3.23).—For the manufacture of a rubberised fibrous composition, rubber solution, after treatment with hydrogen sulphide and sulphur dioxide, is diluted before gelation, and is then mixed with loose fibrous material and with agents for the

precipitation of the rubber on the fibres. The fibres are then removed from the liquid and allowed to gel. D. F. Twiss.

Combining halogen-containing materials with rubber etc. C. E. Bradley and W. A. Gibbons, Assrs. to Naugatuck Chemical Co. (U.S.P. 1,627,725, 10.5.27. Appl., 20.6.21).—" Chlorinated rubber hydrochloride " is obtained by treating a mixture of rubber and solvent with hydrogen chloride and chlorinating the reaction product. D. F. Twiss.

Reclaiming rubber. M. J. Fessler (U.S.P. 1,626,800, 3.5.27. Appl., 29.1.27).—Old or used rubber stock is comminuted and then treated with tetrahydronaphthalene. D. F. Twiss.

Manufacture of a new jelutong product. S. S. Yates (E.P. 271,329, 30.10.26. Conv., 19.6.26; cf. E.P. 245,405; B., 1926, 555).—Jelutong or pontianac is prepared by coagulation of the latex, *e.g.*, with acetic acid or alum. Drying is then effected until a very low proportion of water remains, *e.g.*, 5% or less, instead of the customary 40% or more. The product is obtained in the form of sheet or compact material which can be transported without objectionable deterioration and needs a minimum of refining. D. F. Twiss.

Manufacture of an age-resisting rubber compound. E. R. Bridgwater and D. H. Powers, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,628,528, 10.5.27. Appl., 3.7.26).—Substances, *e.g.*, *p*-aminophenol, which inhibit the ageing of rubber, can be readily dispersed through it prior to vulcanising if they are first intimately mixed with an equal weight of a finely-divided inorganic substance, *e.g.*, barium sulphate, calcium carbonate, or zinc oxide. T. S. Wheeler.

Preparation of a rubber composition. H. W. Elley, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,624,724, 12.4.27. Appl., 5.5.26).—Ageing of rubber is inhibited by mixing a nitroaminophenol, *e.g.*, *p*-nitro-*o*-aminophenol, with it. T. S. Wheeler.

Vulcanisation of rubber. N. A. Shepard and S. Krall, Assrs. to Firestone Tire & Rubber Co. (Reissue 16,648, 7.6.27, of U.S.P. 1,576,072, 20.6.23).—See B., 1926, 453.

XV.—LEATHER; GLUE.

Mechanism of tanning. K. H. Gustavson (J. Amer. Leather Chem. Assoc., 1927, **22**, 236—243).—Portions of ordinary and deaminised hide powder, respectively, were tanned for 48 hrs. with different basic chromium chloride and sulphate solutions respectively, and also at p_H 5·0—the isoelectric point. The p_H value of each liquor was increased to 5·0 by adding the same amount of sodium formate. The p_H values of the exhausted liquor and the chromium content of the tanned powders were determined. The chromium content was lower in the case of tannage at the isoelectric point for a 50% acid chromium chloride solution and for the 41% and 63% acid chromium sulphate solutions, but higher for the 61·5% acid chromium chloride solution. In the deaminised series, the 41% acid chromium sulphate at ordinary p_H value showed a much greater percentage drop in chromium content than the other chromium salts. The results favour the view that the inhibition of cationic chromium fixation by hide protein possessing a less number of reactive basic groups than ordinary hide protein is connected with the diminished acid combining capacity of the structurally altered hide protein. There are disturbing factors, *e.g.*, the formato-complexes, the effect of which is not known. Experiments with ordinary hide powder, powder treated with a buffer solution of p_H 8·0, and powder tanned with formaldehyde at p_H 8·0, using a 61·5% acid chromium chloride solution, showed greater increase in the rate of chromium fixation by hide powders pretreated at p_H 8·0 whether formaldehyde-tanned or only treated with a buffer solution. Chemical changes involving the structure of the protein and its degree of chemical activation, besides colloidal alterations of the micells, are probably the cause of this increase. D. Woodroffe.

Standardisation of hide powder. III. H. G. Bennett (J. Soc. Leather Trades' Chem., 1927, **11**, 87—93; cf. B., 1927, 52).—The alkalinity of hide powders due to organic matter is usually greater in influence than that due to inorganic sources. Determinations of the soluble matter for any one type of manufacture of hide powder yield results which vary concomitantly with the results both of acidity and of alkalinity determinations. Determinations of mineral ash and its alkalinity may or may not vary in the same sense. The absorptive properties of a powder are best indicated by determinations of the soluble matter and its mean variation. The " alkalinity " or " acidity " of the soluble matter is a minor factor. The percentage of ash and its alkalinity are indications of adsorptive power only as they influence the soluble matters present. The alkalinity of the powder should be determined by observing the change in p_H caused by the infusion of 6·5 g. of hide powder in 100 c.c. of saturated boric acid solution, and by titrating the alkalis extracted from hide powder by three successive infusions in saturated boric acid. The results must be related both to the weight of the powder and to the weight of the soluble matter determined. Experiments with two hide powders from the same source showed that high non-tans were obtained with the powder containing a higher proportion of soluble matter and having a higher acidity and alkalinity. The latter are due to amphoteric, soluble organic constituents. D. Woodroffe.

Use of sulphite-cellulose extract as a tanning material. E. L. Wallace and R. C. Bowker (U.S. Bur. Standards, 1927, Tech. Paper No. 339, 309—321).—Experiments have shown that sulphite-cellulose extracts contain a satisfactory amount of material absorbable and fixed by hide powder. They compare favourably with vegetable tanning extracts in these respects. They may be blended with ordinary vegetable tanning extracts without loss of tannin content, and give a satisfactory colour on leather used either alone or in mixtures. They brighten the colour of most tanning materials on leather, moderate the red colour of quebracho and mangrove, and show a marked brightening effect on chestnut extract. They solubilise some of the quebracho insoluble matter. Pieces of hide pre-tanned in sulphite-cellulose liquors of ½%, 1%, and 1½% tannin content

and finished in ordinary mixed tanning extracts required 28 days only as compared with 40 days for hides not pre-tanned. Blends of equal parts of sulphite-cellulose extract, chestnut, and mangrove gave as good a tannage in 35 days as required 40 days with oak bark extract. The chemical, physical, and ageing properties of the leather tanned with sulphite-cellulose extracts compared favourably with those of leathers tanned without their use. D. Woodroffe.

Unhairing action of stannous salts. H. B. Merrill (J. Amer. Leather Chem. Assoc., 1927, **22**, 230—236).—Three samples of hair were treated with saturated lime water, 1% stannous chloride for 24 hrs. followed by lime water for 24 hrs., and 1% stannous chloride solution for 48 hrs. respectively. The lime water alone and stannous chloride solution alone had little action on the hair, but the combined treatment of stannous chloride solution followed by lime water completely disintegrated the hair. Stannic chloride had no such effect. The stannous chloride can be used instead of sodium sulphide and in similar quantity for sharpening milk of lime for loosening the hair on raw skin. The effect of the stannous chloride is attributed to its reducing properties. It acts on hair only in alkaline solution. Its action confirms the theory of Atkin and Thompson (Leather Trades' Year Book, 1926, 56) that sulphides reduce cystine to cysteine, which latter acts as an oxygen carrier, thus oxidising the proteins of the hair and epidermis.

D. Woodroffe.

Patents.

Manufacture of ligno-tanning material from waste sulphite liquor. V. Drewsen, Assr. to West Virginia Pulp & Paper Co. (U.S.P. 1,629,448, 17.5.27. Appl., 14.2.19. Cf. U.S.P. 1,303,176—7; B., 1919, 507 a).—Waste sulphite-cellulose liquor is heated at 130° under pressure with 2% of calcium oxide for about 1 hr. The liquor is then treated with carbon dioxide, filtered, and concentrated, and sufficient sulphuric acid to precipitate the calcium present as calcium sulphate is added. The filtrate is of value as a tanning agent.

T. S. Wheeler.

Solution for depilating hides. H. Benfey (G.P. 436,149, 5.12.24. Cf. E.P. 246,114; B., 1926, 505).—Sodium hydrosulphide solution, *d* 1·22, containing 12·6% of active sulphur, is obtained by scrubbing waste gases from the manufacture of carbon disulphide with a mixture of sodium sulphide solution, *d* 1·24, and milk of lime, *d* 1·16, and subsequently allowing the calcium hydroxide sludge to settle. L. A. Coles.

Leaching apparatus (U.S.P. 1,628,787).—See I.

XVI.—AGRICULTURE.

Effect of soil micro-organisms on paraffin used as a coating to decrease the injurious action of lead arsenate on plant roots. W. E. Fleming (J. Agric. Res., 1927, **34**, 335—338).—Soil micro-organisms, especially various species of fungi, are able to decompose paraffin in the soil and convert it into organic acids. The growth of many soil fungi was markedly stimulated in the presence of paraffin, whether added alone or as a coating on particles of lead arsenate. This would explain the results of Leach (cf. B., 1926, 840), who found that the injury to plants caused by lead arsenate, applied to the soil as an insecticide, was not reduced when the particles were coated with paraffin.

C. T. Gimingham.

Determination of organic matter in soils by means of hydrogen peroxide. W. O. Robinson (J. Agric. Res., 1927, **34**, 339—356).—A method is described for the determination of organic matter in soils by the loss in weight on digestion with hydrogen peroxide. About 1 g. of soil is digested on the water-bath with 10 c.c. of water and 10 c.c. of 30% hydrogen peroxide until bubbles cease to be evolved. The liquid is then filtered through asbestos in a Gooch crucible, and the filtrate and washings are evaporated to dryness, ignited, and weighed. The insoluble matter on the pad is dried for 18 hrs. at 110°. The difference in weight between the water-free soil and the dried residue plus the ash from the filtrate, is taken as organic matter. In some soils an appreciable amount of organic matter is unattacked by hydrogen peroxide; in certain cases this consists mainly of charcoal. Graphite is not attacked; charcoal and coal are partially decomposed. The method tends to give low results, but it is on the whole about as accurate as the combustion method, and, in special cases, much more accurate. It is not applicable to soils high in calcium carbonate, manganese dioxide, or chromium sesquioxide. Various forms of organic matter, including cellulose, humus, and lignite are almost completely destroyed by hydrogen peroxide in the presence of soil, and the method does not determine any clearly defined kind of soil organic matter. The author does not therefore agree that hydrogen peroxide can be used to differentiate between humified and non-humified material, as proposed by G. W. Robinson and Jones (cf. B, 1925, 140).

C. T. Gimingham.

Alkaline and neutral permanganate methods [for nitrogen determination]: comparison of results on raw materials and fertiliser mixtures. H. C. Moore and R. White (J. Assoc. Off. Agric. Chem., 1927, **10**, 202—216).—The results of over 2000 determinations of the " activity " of fertilisers by the alkaline and neutral permanganate methods are summarised, and attention is drawn to the discrepancies. Both methods are empirical, and certain points in the official regulations need to be made more explicit to ensure greater uniformity. The following possible sources of variation are indicated. Samples should be ground just fine enough to pass a 1-mm. sieve. The exact amount of material should be taken in determining water-soluble nitrogen, so that 50 mg. of water-insoluble nitrogen are left in the residue. In the alkaline method the distillation should be carried out in a definite time, *e.g.*, 1 hr., and exactly 95 c.c. of distillate should be collected. The addition of paraffin to prevent frothing leads to a reduction of some permanganate; the use of glass beads is preferable. In the neutral method, complete preliminary extraction with water is essential, as some of the water-soluble nitrogen may be rendered insoluble by the subsequent permanganate digestion. The complete methods embodying these modifications are described. H. J. Dowden.

Modification of the salicylic–thiosulphate method suitable for determination of total nitrogen in plants, plant solutions, and soil extracts. E. R. RANKER (J. Assoc. Off. Agric. Chem., 1927, **10**, 230—251).—The salicylic–thiosulphate method of determining total nitrogen in fertilisers, which is based on the reduction of nitrates in acid medium, was found to give low results when applied to plants and plant materials, recoveries of 27—68% being secured. By testing the vapours for nitrates, nitrites, and ammonia at each individual stage of the determination, the operations in which losses of nitrogen may occur have been detected, and modifications of the procedure to counteract these losses are suggested. The sample must first be rendered neutral or faintly alkaline, by addition of a predetermined quantity of acid or alkali and not by colorimetric adjustment in the presence of an indicator, since the latter method leads to loss of nitrogen during the subsequent evaporation. If water is present, the sample must be evaporated just to dryness under reduced pressure, on a waterbath. Evaporation to ash dryness may in some cases cause loss of nitrogen during the drying, whilst incomplete evaporation will involve losses during the addition of the acid and during the subsequent digestion. The method as thus modified showed small limits of error when compared with the Devarda alloy method. H. J. DOWDEN.

Determination of carbon and nitrogen on the same soil sample. B. E. BROWN (Ind. Eng. Chem., 1927, **19**, 629—630).—The determination is made with fair accuracy by the wet-combustion method. After the carbon has been converted into carbon dioxide by the action of the chromic–sulphuric acid mixture, the nitrogen is present in the residue as ammonium sulphate, and is determined as ammonia in the usual way. Chromic acid gives more satisfactory results than mercuric oxide or manganese dioxide. W. J. POWELL.

Inaccuracies of the Devarda method when applied to plant materials. E. R. RANKER (J. Assoc. Off. Agric. Chem., 1927, **10**, 252—256).—The Devarda method for determining nitrate-nitrogen, although extremely accurate for soils when applied to plant extracts and biological materials, often gives rise to erroneous results. Samples were selected which had been proved free from nitrates, nitrites, and ammonia by tests with diphenylamine and with Nessler's solution. Out of 24 samples, 15, including materials such as sugar cane water-extract, germinated pea seed, carbamide, and glycine, gave a positive nitrate nitrogen determination, although none was present. To avoid incorrect deductions from these results, it is recommended that nitrogen determined by this method should be described as "Devarda nitrogen" rather than "nitrate-nitrogen." H. J. DOWDEN.

PATENTS.

Manufacture of materials serving both as means for destroying plant pests and as fertilisers. M. LANGE (G.P. 438,006, 10.12.20).—The materials consist of solutions of nitrated hydrocarbons or phenols in sulphuric acid to which phosphates are added. *E.g.*, sulphuric acid, containing 10% of its weight of nitrated hydrocarbons, or phenols or their homologues, is mixed with phosphorite in the proportions required to render the phosphate soluble. A distributable powder is obtained, which, in use, does not harm plant roots. C. T. GIMINGHAM.

Fertiliser. E. CERASOLI (E.P. 270,957, 16.10.26).—Residues from the distillation of beetroot molasses are mixed with kieselguhr and phosphorite or calcium sulphate at ordinary temperature. C. T. GIMINGHAM.

Insecticide composition. O. F. HEDENBURG and D. S. PRATT, Assrs. to TOLEDO REX SPRAY CO. (U.S.P. 1,627,389, 3.5.27. Appl., 1.7.19).—An insecticide comprises lead arsenate and an emulsoid colloid, such as gum arabic, mixed with lime-sulphur. C. T. GIMINGHAM.

Manufacture of calcium arsenate [insecticide] products. W. J. LIIPFERT, Assr. to GENERAL CHEMICAL CO. (U.S.P. 1,626,942, 3.5.27. Appl., 6.2.24).—An aqueous suspension of calcium hydroxide is treated under agitation at below 38° with arsenic acid solution, and the paste obtained is heated at the b.p. and dried. T. S. WHEELER.

Manufacture of an insecticide. W. C. PIVER (U.S.P. 1,626,872, 3.5.27. Appl., 30.3.23).—Arsenic acid is treated with sufficient excess of calcium oxide to yield a dry product, and with 0·5—5% of aluminium sulphate. T. S. WHEELER.

Solutions for destroying animal and plant pests. F. HOFFMANN-LA ROCHE & CO. A.-G. (Swiss P. 114,438, 14.11.24).—The solutions contain nicotine soaps of the higher fatty acids, resin acids, or naphthenic acids. L. A. COLES.

Material for destroying animal and plant pests. FARBENFABR. VORM. F. BAYER & CO. (F.P. 595,974, 31.3.25. Conv., 10.4.24).—Copper compounds of aromatic bases, such as aniline and its derivatives and homologues, prepared by treating the bases with copper sulphate solution, are used for the purpose. L. A. COLES.

XVII.—SUGARS; STARCHES; GUMS.

Refining qualities of raw [cane] sugars. T. B. WAYNE (Int. Sugar J., 1927, **29**, 94—100).—It is fallacious to grade raw cane sugars for their refining value strictly on polarisation, as this basis of purchase does not differentiate between the superior refining qualities of some sugars and the inferior nature of others. Characteristics of a good raw sugar are that the crystals shall be uniform, hard, and sharp, having an average diameter of about 0·8 mm., with few needle-shaped crystals. Viewed by projection of their images on a screen, the crystals should be regular and of good shape; the colour value by spectrophotometric analysis at a wave-length of 560 μ expressed as — log (sp. transmissivity), should not exceed 0·7500. With a total colour value of less than 0·7500, the higher the absorption trend at 440μ, the more suitable the sugar is for refining purposes, since this indicates that the colouring matter present is due to caramelisation of an incipient nature, the absence of greyish colouring matter indicating improper clarification of ferric iron–polyphenol compounds. Polarisation to be 96° or more, ash at a maximum of 750° F. not exceeding 0·45%, and moisture by drying *in vacuo* at 80° to constant weight to allow a safety factor of 0·25. J. P. OGILVIE.

Evaporating and heating systems in cane [sugar] factories. G. W. Connon (Int. Sugar J., 1927, 29, 100—103).—Following beet factory practice, cane houses, especially in Cuba, are now following the "extra steam" system for heating the raw juice, this method reducing the total fuel consumption about 4½%. A development of this method is the use of the "dead-end" double effect, the first cell of the evaporator consisting actually of two parts, the first operated with exhaust steam, and the second sending all its vapour to the juice-heaters. There are no connexions on the steam or vapour side between these evaporators and the quadruple effect, but the partly concentrated juice from the dead-end evaporator is sent to the multiple effect to be made into syrup. In this way 8½% less steam is used, compared with the ordinary quadruple effect, the total heating surface being no greater. A number of the larger and more efficient factories in Cuba employ the pre-evaporator or Pauly cell, using live steam at 40 lb. pressure, the evaporation being discharged into the exhaust steam main. A very high rate of evaporation can be obtained, and this system provides the cheapest way to increase the capacity and economy of the evaporation department of a cane factory. One of the factories in Cuba now operates an evaporator employing thermo-compression, with a good economy in its bagasse consumption. J. P. Ogilvie.

Sugar-house incrustations. J. W. Schlegel and J. P. Manley (Ind. Eng. Chem., 1927, 19, 219—221).—A heavy sticky sludge forming on the upper surfaces of the char filters was found to consist mainly of silica, which had probably originated from the use of diatomaceous earth in clarification. Scales found in the outlet pipes from the low-grade char filters were found to have a similar composition. A deposit inside the flue of a char kiln was found to consist essentially of ammonium and calcium sulphates. Scale deposited on the corrugations and faces of the plates of ordinary plate-and-frame presses used for filtering sugar liquors were found to consist mainly of CaO, 21·33; Fe_2O_3 and Al_2O_3 12·41; SiO_2, 14·89; P_2O_5, 7·25; MgO, 6·48; CO_3 6·50; and SO_3, 0·91%. Examination of yellowish-brown stalactitic deposits around the caps of water-tube boilers, caused by leakage from within the boilers, showed that traces of waste-waters from the char filters had leaked into the boilers where it had interacted with the soda ash present. Analyses were also made of the scales found in the Hoppes water purifier, and of sludge taken from a return steam-trap. J. P. Ogilvie.

Beet dehydration process. B. J. Owen, L. F. Manés, and J. L. Dougan (J. Min. Agric., 1927, 33, 986—996).—Cossettes are dehydrated in a single operation (without the preliminary drying advocated by de Vecchis) by a drying agent, *e.g.*, heated air, continuously and uniformly applied until their temperature is raised to about 93°, and the water content has been reduced to about 5% of the weight of the product. This drying may be effected either in mass or in layers, and plant is described for doing so on the large scale in field or factory. An extraction apparatus for exhausting the dried cossettes is employed by means of which the sugar may be obtained in the form of a dense syrup in continuous and expeditious manner, this being in essence a vertical vessel to which the material is fed continuously and impelled upwards, being exhausted by means of a counter-current of water caused to gravitate through the ascending mass. In purifying this dense syrup, it is treated with phosphoric acid, heated at 80°, and limed to neutrality; or it may be heated, centrifuged, and treated with a solution of phosphoric or sulphurous acid, followed by a suitable quantity of dry tricalcium sucrate or dry calcium hydrate, the alkalinity later being adjusted by means of phosphoric acid. This method ensures that the syrup can be filtered under comparatively low pressure. J. P. Ogilvie.

Determination of phosphates in sugar-cane juice. H. B. Springer and J. G. Davies (J.S.C.I., 1927, 46, 143—144 T).—In determining the phosphate content of cane juice as a guide to the manurial requirements of the soil (cf. Walker; B., 1923, 322 A), a modification of the Pemberton–Neumann molybdate method is used, which is stated to give rapid and reasonably accurate results, even in the presence of the usual amounts of silicate occurring in cane juice. The uranium acetate procedure followed by Walker gives results lower than the truth. J. P. Ogilvie.

Determination of sugar in the beet and the use of the Krüger method. A. Le Docte (Int. Sugar J., 1927, 29, 214—218).—The Krüger method is considered unreliable because the volume of liquid added to 26 g. of beet pulp is too small to obtain a perfect admixture and a complete diffusion of the sugar within a reasonable time, and is also too small to obtain rapidly a sufficient quantity of filtrate for use with the Pellet continuous polarimetric tube. Compared with the Sachs and Le Docte procedure, the method is open to criticism on the grounds that it is less rapid and more liable to error in factory routine operation. J. P. Ogilvie.

Hydrogen-ion concentration and the defecation of cane juice. H. S. Paine and R. T. Balch (La. Planter, 1927, 73, 127—132, 148—150).—Provided that sufficient filtration capacity be available, the procedure to ensure a maximum clarification and to prevent inversion losses is to lime to p_H 8—9, measured either before or after heating, but the hydrogen-ion concentration for the maximum defecation varies with different juices, and in any case the possibilities of colour formation and of scaling of the tubes of evaporators and heaters are to be considered. J. P. Ogilvie.

Use of hyposulphites in the [beet] sugar factory. M. Mestre (Bull. Assoc. Chim. Sucr., 1926, 44, 72—79).—Treatment of the juices and syrups with hyposulphites prevents the oxidation of insoluble gelatinous compounds and of soluble metapectic compounds into substances that considerably add to the viscosity, and retard boiling and crystallisation. J. P. Ogilvie.

Use of chlorine for [beet] juice purification. O. Spengler and R. Weidenhagen (Z. Ver. deut. Zuckerind., 1927, 119—121).—A comparison of the results obtained by Ochi's process (B., 1926, 559) with the usual method of clarification showed that on treatment of the juice with chlorine followed by 2% of "Norit," the purity was 90·8°; whereas with 2·5% of lime in ordinary carbonation it was 94·2°. In order to reach a purity

of 95°, it was necessary to use 5% of the decolorising carbon. In applying the process to the purification of beet molasses, it was not possible to effect a sufficient incorporation of chlorine with the dense liquid.

J. P. Ogilvie.

Error caused by the presence of invert sugar in the determination of sucrose in the beet by the aqueous digestion method. V. Staněk and J. Vondrák (Z. Zuckerind. Czechoslov., 1927, **51**, 220—224).—Under the influence of the basic lead acetate in the hot aqueous digestion method, the invert sugar which may be present suffers a loss of its lævo-rotatory power, and may even become dextro-rotatory, its cupric reducing power only slightly diminishing. If the invert sugar content is considerable, it is not exact to correct the polarisation by increasing it by one third of the invert sugar present. It is then necessary to determine the sucrose by double polarisation, or else to ascertain the change of polarisation of the invert sugar present caused by sodium hydroxide at the temperature of operation. J. P. Ogilvie.

Patents.

Extraction of juice from sugar cane [by milling]. W. H. Morgan, sen. (E.P. 267,234, 14.12.25).—Each cane stalk is divided into short sections which are disintegrated longitudinally during their movement between relatively movable blades to produce a long-fibred, finely-shredded absorbent mass, the juice being finally removed from it by pressure or by diffusion.

J. P. Ogilvie.

Preparation of sugar cane stalks prior to the extraction of juice therefrom. W. H. Morgan (E.P. 267,235, 14.12.25).—Cane stalks are prepared for juice extraction (by milling or otherwise) by removing extraneous matter by washing, cutting the stalks into short-length sections, shredding, and reducing them to a clean fibrous hay-like mass containing the juice. The removal of the extraneous matter is effected during the movement of the stalks in a liquid bath in which they are subjected to attrition. J. P. Ogilvie.

Apparatus for concentrating and evaporating liquids, such as syrups. Baker Perkins, Ltd., G. R. Baker, W. E. Prescott, and Soc. Anon. Anc. Etabl. A. Savy Jeanjeon & Cie. (E.P. 268,437, 1.1.26).—The liquid passes through an element containing a spiral passage of gradually increasing size formed between two concentric tubes, the outer of which is heated and the inner serving for the passage upwards of the evolved steam. Heating may be effected by gas, oil-firing, electricity, or other direct means. J. P. Ogilvie.

Recovering sugar content in cane juices. C. G. Petree, Assr. to Petree & Dorr Engineers, Inc. (U.S.P. 1,625,680, 19.4.27. Appl., 20.4.25. Conv., 18.8.24).—Treatment of the rich and weaker juices coming from different stages of a cane-milling circuit consists in decanting the rich juice, adding the mud produced to the weaker juice, decanting the latter, and separately recovering the solids and juice from the residual mud by means of a filter press. J. P. Ogilvie.

Extraction of the sugar present in [beet] molasses and vinasses. H. Friedrich and W. Rajtora (G.P. 435,515, 23.4.24. Conv., 17.5.23).—Thickened molasses or vinasse at about 40° is mixed with glacial acetic acid, and the precipitated sugar separated and dried with hot air, the acetic acid being recovered from the residues by distillation. J. P. Ogilvie.

Decolorisation of [beet] sugar juices. L. J. Heuclin (F.P. 608,097, 19.12.25).—Ozone is applied for decolorising at two stages, viz., at the end of the last saturation, and before, during, or after concentration.

J. P. Ogilvie.

Treatment of [soft] sugar. H. C. Welles (U.S.P. 1,624,296, 12.4.27. Appl., 1.10.23).—The colour of soft (brown) sugar is improved by treatment in the centrifuge with a small quantity (0·1%) of 20% phosphoric acid solution. T. S. Wheeler.

Fertiliser (E.P. 270,957).—See XVI.

XVIII.—FERMENTATION INDUSTRIES.

Briquette binder. Moore and Myers.—See II.

Patents.

Treatment and preparation of yeast and product. F. M. Hildebrandt and C. N. Frey, Assrs. to Fleischmann Co. (U.S.P. 1,625,121, 19.4.27. Appl., 4.6.21).—Yeast is submitted to a conditioning fermentation in a nutrient sugar solution, and is then mixed with about half its weight of maize meal, and of a saturated solution of calcium sulphate, and the mixture is slowly dried in a current of air at 17° until the moisture content is reduced to about 13%. T. S. Wheeler.

Stabilisation of yeast. F. Sauer (G.P. 436,878, 21.1.25).—Pure yeast cultures are treated with weakly acid salt solutions, the strength and composition of which correspond with the salt content of the yeast plasma, and stabilised by the addition of substances such as alcohol or glycerol which do not hinder the growth of the yeast cells. A. R. Powell.

Production of potassium bitartrate from grape residues. C. Spedicato (F.P. 602,243, 7.8.25).—The residues are stirred with sodium carbonate and treated with live steam for 20 min. in a vessel provided with a perforated false bottom, the liquid collecting below this being subsequently treated with concentrated sulphuric acid to precipitate potassium bitartrate. L. A. Coles.

Fermentation processes for the production of butyl alcohol and acetone. Commercial Solvents Corp., Assees. of D. A. Legg (E.P. 249,833, 25.2.26. Conv., 30.3.25).—See U.S.P. 1,582,408; B., 1926, 563.

XIX.—FOODS.

Preparation and analysis of the various proteins of wheat flour with special reference to the globulin, albumin, and proteose fractions. W. F. Hoffman and R. A. Gortner (Cereal Chem., 1927, **4**, 221—229).—Details are given of the methods of isolation from a patent wheat flour of gliadin, glutenin, leucosin, and small amounts of a possible globulin, together with the results of the protein analysis by the Van Slyke method (cf. B., 1911, 1135). No proteose could be detected, but a gum was obtained which, in a state of purity, did not colour blue with iodine or reduce Fehling's solution before hydrolysis. Extraction of wheat flour with 5% potassium sulphate solution and with 10% sodium

chloride solution gave very different results, both as regards the nature and amount of the proteins which could be separated from the solutions. F. R. ENNOS.

Concentration of glutenin and other proteins in various types of wheat flour. E. GREWE and C. H. BAILEY (Cereal Chem., 1927, 4, 230—247).—Determinations of glutenin by the method of Sharp and Gortner (B., 1924, 762) and by that of Blish and Sandstedt, for a number of flour samples milled from wheat of varying types, gave results in satisfactory agreement, but the relative baking qualities as judged by the volume of the loaves obtained in three different baking tests were not consistent. The ratio of glutenin to crude protein and of glutenin to the sum of glutenin and gliadin showed very little variation over the whole range of flours examined. The highest coefficient of correlation with loaf volume was shown by the crude protein content and the extensibility, and a lower one by the diastatic activity of the flour. No correlation was shown by the quality constant (Sharp and Gortner, B., 1924, 29), the size of the starch granules, or the heat of imbibition of the dried starch preparations. F. R. ENNOS.

Determination of casein in milk by approximately isoelectric precipitation. H. C. WATERMAN (J. Assoc. Off. Agric. Chem., 1927, 10, 259—263).—For the determination, a buffer solution of p_H 4·7 is used. The optimum point is p_H 4·74 as shown by Rona and Michaelis (cf. A., 1910, i, 905). The buffer precipitant is prepared from 250 c.c. of *N*-acetic acid to which are added 125 c.c. of *N*-sodium hydroxide solution, the mixture being diluted to 1 litre. To avoid the tedious washing of the casein precipitate, with its inherent losses, casein nitrogen is determined as the difference between the total nitrogen in the milk and the nitrogen content of the filtrate after precipitation. To 20 c.c. of the milk sample add 50 c.c. of the buffer solution, dilute to 100 c.c., and, after shaking, warm at 50—60° for 15 min. Cool, filter (returning the filtrate twice), and determine both nitrogen (A) in 50 c.c. of the filtrate and total nitrogen (B) in 10 c.c. of the original milk. Then $6 \cdot 38 \times (B - A)$ = grams of casein in 10 c.c. of the milk. H. J. DOWDEN.

Morres and Schützler's method for the determination of fat in milk. O. BAUMANN (Pharm. Zentr., 1927, 68, 289—290).—Comparative experiments by the new method (Morsin method) and by the Gottlieb–Röse and Gerber methods show that it is entirely reliable, even in presence of formaldehyde, for milks of which the acidity is below 20 on the Soxhlet-Henkel scale. With higher acidities, or after boiling, the separation of the fat is incomplete, but pasteurised milks give accurate results. The method is very rapid and convenient. S. I. LEVY.

Determination of nitrate- and nitrite-nitrogen in meat. O. NOETZEL (Pharm. Zentr., 1927, 68, 321—323).—10 g. of the finely-divided meat and 150 c.c. of water containing a few drops of 25% sodium hydroxide are frequently shaken over a period of about 1½ hrs. After bulking to 200 c.c., 150 c.c. of the filtrate are treated drop by drop with 30 c.c. of dialysed iron solution, diluted to 200 c.c., shaken vigorously, and filtered. One portion (40—80 c.c.) of the filtrate, which should be quite clear, is used for the determination of total nitrogen by adding a slight excess of 0·1*N*-potassium permanganate solution, acidifying with sulphuric acid, destroying the excess of permanganate with alcohol, and boiling for 2 min. after the addition of 1 g. of powdered iron. 9 c.c. of 1 : 2 sodium hydroxide solution are added to the cooled solution and the ammonia steam-distilled into acid, the excess of which is titrated using Congo-red indicator. In the other portion of the filtrate nitrite is determined colorimetrically by means of *m*-phenylenediamine hydrochloride. The method is very satisfactory for the determination of nitrite in the presence of relatively large quantities of nitrate, but not *vice versa*. F. R. ENNOS.

Determination of benzoic acid in minced meat. E. WALTZINGER (Chem.-Ztg., 1927, 51, 170).—To 20 g. of the minced meat shaken with water are added 20 c.c. of Fehling's copper sulphate solution, and 6 c.c. of *N*-sodium hydroxide solution, and the process continued as in the determination of benzoic acid in egg yolk (B., 1927, 122). The extract obtained from meat with ether and light petroleum may contain other substances, such as fatty acids, especially if putrefaction has set in. The extract is weighed, heated at 100° to sublime the benzoic acid, and weighed again. With perfectly fresh meat titration of the extract with 0·1*N*-sodium hydroxide solution gives satisfactory results. F. R. ENNOS.

Heat and moisture as factors in the destruction of gossypol in cottonseed products. W. D. GALLUP (Ind. Eng. Chem., 1927, 19, 726—728).—Gossypol, the toxic principle of cotton seeds (cf. Carruth, A., 1918, i, 266), is readily changed on heating to the less toxic *d*-gossypol (cf. Sherwood ; B., 1926, 564), which is slowly destroyed, but the time of heating necessary to obtain a non-toxic product is much longer than that employed in the oil mill. Heating the seeds with water in an autoclave at 20 lb. pressure destroys both the gossypol and *d*-gossypol in relatively short periods of time (1—4 hrs.), and feeding tests show that the product thus obtained yields a more suitable cattle food than the untreated seeds. Germination of the seeds does not decrease the toxicity. W. J. POWELL.

Aluminium cans for preserving foods. H. SERGER (Chem.-Ztg., 1927, 51, 391—393, 411—413).—Tests with a large number of fruits and vegetables show that they may be advantageously preserved in aluminium cans without suffering any deterioration as regards taste, colour, odour, or edibility. The aluminium is much less attacked by the fruit acids than is the tin of the usual tinned-iron cans, and any minute amount of aluminium that dissolves has no toxic effect. A. R. POWELL.

Oils in emulsions. BALDWIN. See II.

PATENTS.

Extraction of albumino-caseins of vegetable origin, and separation of these albumins from amylaceous matters. H. BEAUFOUR (E.P. 260,242, 12.10.26. Conv., 23.10.25).—Vegetable substances such

as cereals or oleaginous seeds are treated below 50° with a 3—10% solution of a chloride of an alkali or alkaline earth, the albuminous solution being separated from the insoluble material. After coagulation by suitable means, the albumino-casein is collected by decantation, filtering, or centrifuging and is washed, dried, and bleached. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 147,513, 121,141, 17,116 of 1906, 9457 of 1905, 30,842 of 1897, and 25,786 of 1896.]

F. R. Ennos.

Preparation of concentrated vitamin-*B*. P. Caccia (U.S.P. 1,617,702, 15.2.27. Appl., 25.9.25).—A mixture of yeast and water is kept for several days and filtered. The residue is extracted with boiling dilute hydrochloric acid, and the extract mixed with the filtrate and evaporated to a paste, which is extracted with absolute alcohol. The alcoholic extract is treated with ethylene chloride to precipitate vitamin-*B*, which is recrystallised from sulphuric acid. T. S. Wheeler.

Deodorising of fluids (E.P. 270,509).—See I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Cheap ethylene dichloride. D. H. Killeffer (Ind. Eng. Chem., 1927, **19**, 636—639).—The commercial possibilities of ethylene dichloride are examined. This substance is now cheaper than carbon tetrachloride on a volumetric basis of comparison, and is suitable for use as a non-inflammable solvent since it has a relatively low sp. heat, forms explosive mixtures with air only with difficulty, and will not support a flame. It is superior to carbon tetrachloride in stability towards water and steam, hence is less liable to cause corrosion of plant. The sharp reduction in price of ethylene dichloride has also made possible its utilisation as a raw material for synthetic products, since under suitable conditions both chlorine atoms are replaceable by other groups. With benzene it yields dibenzyl, which is readily oxidised by atmospheric air to benzaldehyde or benzoic acid, and with ammonia yields ethylenediamine. Glycol diacetate, a solvent for cellulose acetate and nitrate, can be made by heating ethylene dichloride with a small excess of anhydrous sodium acetate under 125—150 lb. pressure. The addition of about 5% of glycol diacetate to the original mixture furnishes a common solvent and hence accelerates the action, whilst excess of sodium acetate is necessary to prevent the formation of by-products. Succinic acid can be prepared without difficulty by the condensation of ethylene dichloride with sodium cyanide, the succinonitrile formed being hydrolysed by means of hydrochloric acid. W. J. Powell.

Application of the Stahre reaction to the accurate determination of citric acid. B. G. Hartmann and F. Hillig (J. Assoc. Off. Agric. Chem., 1927, **10**, 264—272).—The determination of citric acid by oxidation to acetone and precipitation as pentabromoacetone (cf. Wöhlk; B., 1902, 564) is quantitative if certain precautions are taken. The solubility of the pentabromo-compound is the chief source of error, and must be corrected for by allowing 1·7 mg. for every 100 c.c. of reaction mixture. The tendency to form a difficultly crystallisable oil is inhibited by adding asbestos and cooling in a refrigerator before addition of the ferrous sulphate solution. The precipitate is most conveniently dried by aspirating air. To 100 c.c. of citric acid solution add exactly 10 c.c. of dilute sulphuric acid (1 : 1) and 10 c.c. of saturated bromine water, and filter after 10 min., if necessary. To 100 c.c. of the filtrate add 5 c.c. of potassium bromide solution (15 g. in 40 c.c. of water) and a little asbestos, heat at 48—50° for 5 min., and add sufficient 5% potassium permanganate solution to render the supernatant liquid a brown colour, about 15 c.c. being required. Cool in ice-water, and, after adding sufficient ice-cold ferrous sulphate solution (20 g. in 100 c.c. of water containing 1 c.c. of sulphuric acid) to dissolve all the manganese dioxide, about 40 c.c. being required, shake for 5 min., and place in a refrigerator overnight. Filter by decantation, wash with cold very dilute sulphuric acid and with water, dry by aspirating air through the filter, and weigh. Remove the pentabromoacetone from the filter by washing with alcohol and then with ether, and re-weigh. After correcting for the solubility of the pentabromo-compound, the conversion factor 0·424 is used.

H. J. Dowden.

Determination of terpin hydrate in terpin hydrate elixir. A. G. Murray (J. Assoc. Off. Agric. Chem., 1927, **10**, 257—259).—Owing to the sparing solubility of terpin hydrate in ether or chloroform, the extraction of this substance from aqueous solution by these solvents is long and tedious, but may be facilitated by the addition of salt and a little alcohol. Since the hydrate is efflorescent, it must not be dried in a desiccator, nor must heat be used in evaporation of the solvent. The method suggested consists in adding to a measured volume of the elixir sufficient salt solution (20 g. of sodium chloride in 100 c.c. of water) to reduce the alcohol content to 10—15% by vol. Extract 4 times with chloroform containing 5—7% of alcohol by vol., washing each extract through 5 c.c. of the salt solution. Filter the united extract through cotton wool into a weighed dish, and evaporate the solvent by an air-blast without heating. Expose the residue to the atmosphere for 15 min. before weighing.

H. J. Dowden.

Determination of benzoic acid. Waltzinger.—See XIX.

Patents.

Catalyst for synthetic methyl alcohol production. J. C. Woodruff and G. Bloomfield, Assrs. to Commercial Solvents Corp. (U.S.P. 1,625,924—9, 26.4.27. Appl., [A], 26.5.26; [B], 21.6.26; [C], 25.9.26; [D, E], 14.10.26; [F], 25.10.26).—(A) A catalyst for use in the synthesis of methyl alcohol from hydrogen and carbon monoxide comprises one or more difficultly reducible metal oxides, *e.g.*, zinc oxide, magnesium oxide, cadmium oxide, one or more easily reducible metal oxides, *e.g.*, (C) nickel oxide, (D) cobalt oxide, (E) copper oxide, and a metallic halide, *e.g.*, zinc chloride. (B) The catalyst comprises one or more difficultly reducible group II metal oxides, *e.g.*, zinc oxide, one or more difficultly reducible metal oxides of groups III—VII, *e.g.*, chromium oxide, and a metallic halide. (F) A suitable catalyst can be obtained by evaporating to dryness a suspension of zinc oxide in

an aqueous solution of chromium nitrate, ferric nitrate, and zinc chloride, and heating the product to transform the nitrates to oxides. The mixed gases are passed over it at 420° and 250 atm. T. S. Wheeler.

Dehydration of ethyl alcohol. W., K., L., W., and F. Merck (E. Merck) (E.P. 270,612, 9.2.27. Conv., 30.10.26).—The dehydration of alcohol by lime is accelerated by heating in an autoclave at a pressure greater than atmospheric. A. Davidson.

Concentration of dilute acetic acid. H. Suida (U.S.P. 1,624,812, 12.4.27. Appl., 9.2.26. Conv., 21.2.25. Cf. E.P. 218,271—2; B., 1925, 899).—In the application of the processes described in the patents cited, the cresol removed from the extracting apparatus by water vapour is recovered by keeping the aqueous condensate until most of the cresol separates, and then extracting the aqueous layer with oleic acid, from which the cresol is recovered by distillation *in vacuo*. T. S. Wheeler.

Purification of acetic anhydride. British Celanese, Ltd., and W. P. Skertchly (E.P. 270,809, 13.2.26).—Acetic anhydride containing sulphur or sulphur derivatives is purified by treatment with an anhydrous powdered metallic acetate, then with chlorine in quantity sufficient to react with the sulphur etc., and subsequent distillation. B. Fullman.

Manufacture of acetaldehyde. Consortium für Elektrochem. Ind. G.m.b.H. (E.P. 260,305, 26.10.26. Conv., 26.10.25).—The manufacture of acetaldehyde by the action of acetylene on water in presence of a mercury compound is improved by the presence in the reaction mixture of an organic solvent for acetylene, containing a hydroxyl or carbonyl group, but not a carboxyl group. Mono- and poly-hydric alcohols, phenols, and ketones are suitable, as also are substances capable of yielding them such as acetals and esters. To promote rapid removal of acetaldehyde from the reaction liquid, thus avoiding resinification, a rapid current of acetylene is passed through, whereby also the liquid is suitably agitated and the temperature regulated. By fractional condensation of the vapours, water and solvent are returned to the reaction vessel, while acetaldehyde and excess of acetylene pass through washers which absorb the aldehyde, the acetylene being returned to the reaction vessel. Spent catalyst is removed continuously or at intervals and, after regeneration, *e.g.*, by dissolving the separated mercury in nitric acid and converting into sulphate, returned to the system. The reaction may be carried out in presence or absence of acid and at pressures higher than atmospheric. A. Davidson.

Manufacture of acetals. Consortium für Elektrochem. Ind. G.m.b.H. (E.P. 264,791, 12.10.26. Conv., 25.1.26. Addn. to E.P. 257,622; B., 1927, 379).—The process described in the prior patent may be carried out with alcohols containing water, the water being converted into acetaldehyde. The acetaldehyde is separated as far as possible by washing the vapours or fractionally condensing them. The proportion of acetal in the product is increased by suitable partial condensation of the vapours, the condensate being returned to the reaction mixture, or by passing the products of reaction, after removing the aldehyde, repeatedly through the reaction apparatus. A. Davidson.

Electrolytic oxidation of organic compounds. C. H. Field and G. Owen (E.P. 265,672, 7.11.25).—The electrolytic oxidation of organic compounds, especially of anthracene, is effected in the presence of a catalyst prepared from a cerite ore (composition given) from Riddarhytton, Sweden, containing chiefly: SiO_2, 20%; Ce_2O_3, 22·38%; Di_2O_3, 33·1%; and La_2O_3, 10·64%. The ore is heated at 200° for 8 hrs. with sulphuric acid, and the product when cool treated with cold water, the resultant solution being precipitated with oxalic acid (the precipitate being heated to expel carbon dioxide) or an alkali. The red precipitate as produced is added to the electrolyte in quantities of about 5% by wt. of the solution. The cell may be a lead-lined vessel with lead electrodes, the cathode being covered with a diaphragm of asbestos cloth or earthenware. *E.g.*, 3½ cwt. of anthracene (40% upwards) which has been treated with sodium hydroxide is electrolysed in 2—3½ tons of 50—70% sulphuric acid containing 5% of catalyst, at 100°, 4—7 volts, 300 amp. (*D.C.*), for 2 hrs. A high yield of anthraquinone is obtained. Toluene (to benzaldehyde), *m*-xylene, or naphthalene may also be oxidised. The action of the catalyst is attributed to the presence in the ore of an unisolated element associated with the didymium. B. Fullman.

Catalytic dehydrogenation. I. G. Farbenind. A.-G. (E.P. 262,120, 25.11.26. Conv., 30.11.25).—Zinc sulphide and other sulphides of the heavy metals, either alone or mixed mutually or with other materials, often on a carrier, are used for catalytic dehydrogenation. *E.g.*, *iso*butyl alcohol gives *iso*butaldehyde in 92% yield when passed over zinc sulphide on pumice stone at 460°. B. Fullman.

Potassium bitartrate (F.P. 602,243).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Calibration of photographic plates. F. S. Hogg and C. H. Payne (Nature, 1927, **119**, 925).—Density curves for plates with two different emulsions show no greater differences than those for plates with the same emulsion; also, the two kinds of developer employed give closely similar results. The Harvard individual plate calibration curves allow for changes in sensitivity due to the temperature and humidity during exposure in the telescope, to the ageing of the plates, to delayed development, and to sky fog. A. A. Eldridge.

Chemical sensitisation of silver iodide–gelatin plates. H. Frieser (Phot. Ind., 1927, 520—521).—The preparation of a silver iodide emulsion is described, and results are given of tests of the latent image and print-out effects, using a large number of organic and inorganic sensitisers. W. Clark.

Patents.

Photographic media. H. Wade. From Wadsworth Watch Case Co. (E.P. 270,387, 4.2.26).—A stable light-sensitive varnish consists of a hydrophobic colloid containing metal salts capable of combining with free halogen. The salts are present to a point

approaching saturation, and the free halogen is introduced in such amount as to combine with most of the salts and to avoid the presence of free halogen after the metal halides have been formed. As an additional sensitiser, a halogen-substituted hydrocarbon, such as iodoform, may be added. The varnish consists in the main of a light-sensitive oil such as tung oil, and a smaller amount of a resin, such as coumarone, and of gutta-percha may be added. A solution of iodine may be used as the halogen, and lead resinate, for example, as the metal salt. W. CLARK.

Photographic process [using asphalt]. H. WADE. From WADSWORTH WATCH CASE CO. (E.P. 270,386, 4.2.26).—A sensitive medium for use in the preparation of screenless grained litho-plates, line engravings, half-tone blocks, intaglio blocks, etc. consists of asphalt containing a sensitiser which will liberate a halogen under the influence of light. A suitable sensitiser comprises both a metallic halide and a halogen-substituted hydrocarbon, such as iodoform. One medium is prepared by mixing together approximately 10% of asphalt, 40% of solvent naphtha, 25% of benzine, and about 25% of a solution containing a halogen-liberating compound, *e.g.*, 34% of iodoform and methyl ethyl ketone mixture in the ratio of 12 : 22, and 66% of a solvent such as solvent naphtha. W. CLARK.

Recovery of metals [silver] from solutions. G. J. LEVY, Assr. to HOLLYWOOD CHEMICAL CO. (U.S.P. 1,624,172, 12.4.27. Appl., 23.12.25).—Waste sodium thiosulphate solution containing silver is mixed with zinc hyposulphite solution, when all the silver present is precipitated in the metallic form. T. S. WHEELER.

Recovery of silver from photographic spent thiosulphate solution. E. K. GIFFEN (U.S.P. 1,629,212, 17.5.27. Appl., 19.9.25).—Spent sodium thiosulphate solution containing dissolved silver is employed as electrolyte in a cell containing a copper positive pole and a zinc negative pole, connected through an external resistance. Silver is deposited on the copper pole which is fixed at the bottom of the solution, while the zinc pole, immersed in a porous bag, is maintained at the top, so that the silver deposited cannot be contaminated by hydrogen sulphide evolved at the zinc pole. T. S. WHEELER.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Manufacture of smokeless [flashless] explosive powder. F. I. and E. DU PONT, Assrs. to U.S.F. POWDER CO. (U.S.P. 1,627,691—4, 10.5.27. Appl., [A, B], 3.5.22; [C], 8.8.21; [D], 23.2.21).—(A) A flashless propellant comprises nitrocellulose, gelatinised in presence of finely-divided ammonium nitrate, with or without addition of glyceryl nitrate. The addition of acetone to the ethyl ether–alcohol gelatinising mixture is of advantage when glyceryl nitrate is present. The product is formed into grains perforated sufficiently to bring the loading density 15—50% below that of normal cellulose nitrate powders. (B) Nitrocellulose is ground in presence of water or petrol with 4—25% of a mixture of barium nitrate (2 pts.) and potassium nitrate (1 pt.). The product is dried, treated with a gelatinising mixture of ethyl ether and alcohol, and formed into grains. (C) The mixture of barium and potassium nitrates employed in (B) is replaced by black powder or (D) by a mixture of potassium dichromate and charcoal. T. S. WHEELER.

Manufacture of a smokeless [flashless] explosive powder. Flashless cannon powder. F. I. and E. DU PONT, Assrs. to U.S.F. POWDER CO. (U.S.P. 1,627,638—9, 10.5.27. Appl., [A], 10.11.25; [B], 13.11.26).—(A) The process described in U.S.P. 1,627,692—4 (preceding abstract) is modified, in that the nitrocellulose is ground with the added substance in presence of alcohol, to which ethyl ether is then added to form a gelatinising mixture. (B) In the application of the process described in (A) a mixture of potassium nitrate and charcoal is preferable to black powder if the propellant contains glyceryl nitrate. T. S. WHEELER.

Sodium azide (U.S.P. 1,628,380).—See VII.

XXIII.—SANITATION; WATER PURIFICATION.

Detection and determination of very small quantities of iron in drinking and industrial waters. F. KRÖHNKE (Gas- u. Wasserfach, 1927, 70, 510—511).—A solution of *iso*nitrosoacetophenone in chloroform (1·5 g. in 200 c.c.) is a more sensitive reagent for ferrous iron than alkali thiocyanates for ferric iron, and can be used to determine quantities down to 0·01 mg. per litre. To 50 c.c. of the water to be tested 1 c.c. of a 2% solution of hydrazine sulphate is added, and the solution boiled until reduced in bulk to 10—20 c.c. (10—15 min.). The solution is rapidly cooled and shaken with 1 c.c. of the reagent, a 1—2% solution of ammonia being gradually added until a blue colour appears in the chloroform. The colour is compared with standards prepared from a range of solutions of known iron content (0·01—0·1 mg. per litre, or higher if necessary). The presence of manganese does not interfere with the test, but copper, nickel, and cobalt must be absent. A. B. MANNING.

***o*-Tolidine test for chlorine.** HOPKINS.—See VII.

PATENTS.

Manufacture of disinfecting, deodorising, or sanitising substances. W. & F. WALKER, LTD., and J. T. FREESTONE (E.P. 270,440, 20.2.26).—*p*-Dichlorobenzene, made by chlorinating benzol, separating the monochlorobenzene by distillation and the *o*-dichlorobenzene by crystallisation at low temperatures, is used in the form of compressed blocks. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 19,688 of 1912.] W. G. CAREY.

Insecticide. A.-G. FÜR ANILIN-FABR. (E.P. 253,065, 14.10.25. Conv., 2.6.25).—An insecticide for washing animals consists of a soluble inorganic parasite poison and an insoluble inorganic parasite poison in colloidal form, together with a protective colloid and, if necessary, a wetting agent. The colloidal poison may be replaced by a soluble derivative decomposable by acids or carbon dioxide to liberate the insoluble poison in colloidal form; *e.g.*, sodium sulphoarsenite gradually forms colloidal arsenic sulphides on the animal by the action of atmospheric carbon dioxide. W. G. CAREY.

Filter (U.S.P. 1,630,079).—See I.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 5, 1927.

I.—GENERAL; PLANT; MACHINERY.

Theory of fine grinding. IV. Air analysis of large quantities of crushed sand. G. Martin and W. Watson. **V. Existence and preparation of statistically homogeneous grades of crushed sand.** G. Martin, E. A. Bowes, E. H. Coleman, and T. H. Littlewood (Trans. Ceram. Soc., 1926, **25**, 226—239, 240—257; cf. B., 1926, 903).—IV. Apparatus for elutriating large quantities of crushed sand, with air velocities varying from 2 in. to 20 ft. per sec., is described, The compound interest law connecting particle size and number was confirmed, log W/X^3 plotted against X giving a straight line, where W is the weight of a grade, and X the average arithmetical diameter of particles in a grade. The grading velocities and particle size of various grades of sand were determined. V. By repeated air elutriation, crushed sand can be separated into "homogeneous" grades, in which the average arithmetical diameter of the particles cannot be altered by further fractional elutriation. The shape of the distribution curve obtained by plotting the frequency number of the particles against their diameters also remains unchanged. The average frequency curve obtained by plotting the frequencies against the diameters of the particles in a homogeneous grade is a probability curve. F. Salt.

Particle size and volume of a powder. H. Wolff (Farben-Ztg., 1927, **32**, 1667—1669).—The author corrects the misleading assumption that the particle size of a powder varies inversely as the volume occupied by the powder in close packing. The volume occupied by spheres and cubes of different sizes is mathematically treated from first principles, and it is shown that shape and not size of particle influences the bulk of a closely packed powder, as well as its rate of settling.
S. S. Woolf.

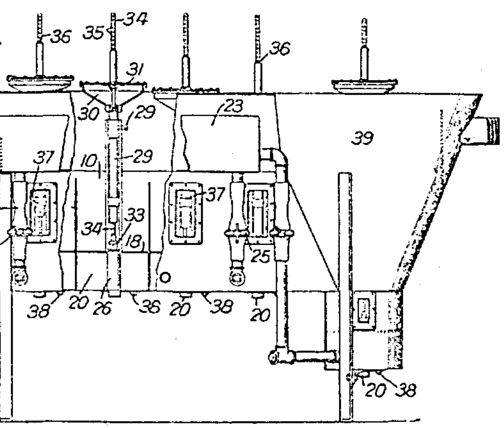
Technical uses of *X*-rays. A. St. John (Ind. Eng. Chem., 1927, **19**, 339—342).—Applications of *X*-rays to industrial problems, such as detection of flaws in engineering materials or of ash in coal, and the determination of atomic structure in carbons, are described.
L. M. Clark.

Colour measurement. T. Oryng (Physikal. Z., 1927, **28**, 298—300).—A review of methods and replies to criticisms. The only unequivocal method of colour measurement depends on troublesome investigations of absorption spectra. Two alternative methods, those of Ostwald and Hübl, are available, but it is concluded that further research is necessary to decide between them. R. A. Morton.

Patents.

[Fahrenwald] hydraulic classifiers. E. C. R. Marks. From Dorr Co. (E.P. 268,663, 4.10.26).—The aim of this invention is the discharge of the coarse or heavy solids at exactly the same rate as they are collected, so that there is always a bed of settled pulp round the outlet valve—settled, but not compacted, because the upward current or hydraulic water passes

FIG. I.



through it. Several classifying compartments may preferably be combined in one structure as shown in the diagram, where the main flow of pulp is from left to right in a longitudinal upper passage and hydraulic water is supplied through the adjacent passage, 23, and pinchcocks, 25, to chamber, 20 (reverting to the singular), beneath the classifying compartment, 10, the division, 18, between these two compartments being perforated with a number of holes of which the spacing is large compared with their diameter. The discharge valve, 33, admits the settled solids to a pipe, 26, which passes through the hydraulic water chamber. The operation of the valve is controlled by the balance of a longer, clear water column within the pipe, 29, against a shorter pulp column in settling chamber, 10, and the main supply channel. Should the solids accumulate, the pulp column becomes heavier and causes the clear water column to rise; this creates pressure upwards on the flexible diaphragm, 31, of the control chamber, 30, and opens the valve, 33, by means of its long stem, 34, and *vice versa*. The whole control unit from outlet pipe, 26, to valve stem, 34, is quickly detachable, and, to compensate for the fact that, in general, settled pulp of finer material in later compartments is less dense than that of coarser material, the height of the diaphragm, 31, is adjustable by sliding the pipe, 29, and securing

it by a set screw, corresponding adjustments to the length of valve stem being effected by pin, 36, in holes, 35. A bleeder, 38, is provided to take care of stray solid matter passing the perforated plate, 18. The final compartment of the series of classifiers shown includes a large conical settling tank, 39, so that very fine solids may be collected. The upper edge of the nearly stationary settled pulp may be observed through the windows, 37; the stream of thick pulp from outlets, 26, also gives visual indication that the apparatus is working properly.

B. M. VENABLES.

Wet-separation of the constituents of mineral and other pulp. T. G. MARTYN (E.P. 264,128, 30.8.26. Conv., 7.1.26).—The pulp is stratified by upward-current hydraulic water, and as the pulp as a whole flows substantially horizontally the various layers are cut off and collected in turn by partitions depending from the closed top of the apparatus, there being a draw-off pipe at the apex between each partition. The stratifying compartment at the bottom is V-shaped in vertical section and contracts away from the feed end. Hydraulic water is supplied through a number of inlets at the bottom of the V.

B. M. VENABLES.

Separation of a mixture of liquids having different boiling points. E. CHUR (E.P. 271,130, 22.1.26).—The liquid is first distilled in a main column in which the vapours and liquid both move downwards; the still may be heated either by steam supplied to the bottom and passing upwards under each tray in succession, or the liquid may be led away from each tray to outside the column, subjected to direct heat in a flue or in a tar still, and sent back to the next lower tray. The vapours from a number of successive sections of the main column are led to separate auxiliary columns (provided with coolers) and the purified vapours collected from the latter. The liquid from one auxiliary column passes to the next auxiliary column in the same order as the original vapours were taken off the main column.

B. M. VENABLES.

Removal or separation from gaseous fluid of material suspended therein. R. S. PORTHAM, and TANGENTIAL DRYERS, LTD. (E.P. 271,545, 17.2. and 12.11.26).—The dusty gas is caused to impinge on a device having a number of points, *e.g.*, a bundle of pointed rods, through the interstices of which the collected dust can flow away, and is then given a circular motion by means of vanes, and while rotating, caused to impinge on a number of bars, or the circular motion may be produced by scrolls which divide the stream of gas into thin laminæ.

B. M. VENABLES.

Manufacture of adsorbents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 271,564, 25.2.26).—Silica or metal oxides, *e.g.*, alumina, are obtained in an active condition from mineral jellies by removing part of the impurities by washing, then drying above 120°, and removing the remaining impurities from the shrunk mass by boiling with acids, and then with water, finally drying above 120°.

W. G. CAREY.

Manufacture of porous artificial [filter] stones. SCHUMACHER'SCHE FABR. G.M.B.H. (E.P. 251,964—5, 24.4.26. Conv., 8.5.25).—(A) A mixture of a synthetic resin with a stony raw material of a definite, substantially uniform granular size is moulded under pressure and heated at 100—200°, forming a porous stone of a predetermined degree of porosity for use in filtering. The granulated material may be fluorspar for a stone proof against hydrofluoric acid, or coke for a stone proof against caustic solutions. (B) A stony raw material is mixed with organic substances, *e.g.*, a phenolic substance and an aldehyde, which form hard insoluble resinous condensation products when heated, with or without the addition of a catalyst, the mixture being moulded under pressure and heated.

B. W. CLARKE.

Low freezing-point solutions. PYRENE CO., LTD., Assees. of A. Z. PEDERSEN (E.P. 267,561, 14.3.27. Conv., 27.5.26).—A solution suitable for motor-car radiators etc. is made by dissolving 35 pts. by wt. of calcium chloride in water, and then mixing with a solution containing 1 pt. by wt. of sodium chromate and neutralising agent, *e.g.*, chromic acid.

W. G. CAREY.

Refrigerating machine. H. SCHIER (E.P. 271,358, 30.12.26).—In a continuously operating absorption refrigerating system the energy of the cooling water delivered under pressure is used to operate the circulating pump for refrigerant, and pressure-controlling devices are arranged between the high- and low-pressure sides of the system to prevent the difference in pressure from rising so far as to stop the circulation.

B. M. VENABLES.

Absorption refrigerating apparatus. D. W. BERLIN (E.P. 271,634, 18.5.26).—In an absorption refrigerator where one vessel acts alternately as boiler and absorber and another vessel as condenser and then as evaporator or refrigerator proper, the two vessels are rigidly connected by twin conduits which are in close relation to each other, and also lead to and from the nearest points on the two vessels, and are so arranged that the whole liquid contents of the refrigerator can be run back into the boiler on tilting the vessels. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 234,110, 16,293 of 1888, and 3153 of 1877.]

B. M. VENABLES.

Kiln. E. L. JOHNSON (E.P. 271,291, 7.8.26).—A shaft kiln is provided with a hearth for solid fuel situated in a tunnel placed diametrically across the bottom of the kiln. Outlets for products of combustion are formed through the walls of the tunnel in such a manner, *e.g.*, inclined first upwards then downwards, that the material in the kiln cannot enter the combustion chamber.

B. M. VENABLES.

Annular ovens or furnaces with rotating hearths for treating pulverulent, granular, or like materials. TROCKNUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-G.M.B.H., L. HONIGMANN, and F. BARTLING (E.P. 271,707, 2.10.26. Addn. to E.P. 259,389; B., 1926, 999).—Modifications to the oven described in the original patent are described, comprising means for depositing a uniform layer of charged material, and a jet and collecting hood for discharging the material.

B. M. VENABLES.

Method and apparatus for annealing metals, ceramic and other materials. A. BESTA (E.P. 248,394, 27.2.26. Conv., 28.2.25).—The articles to be heat-treated are placed in interchangeable boxes or

containers and pushed in succession up one side of a tower, across the top, and down the other side. Strong heating is confined to the upper sections of the tower, and may be effected by reheating a portion of the gases circulating round the upper part only while the other portion of the gases is brought down to the bottom of the tower and recirculated. B. M. Venables.

Grinding mill. P. Hänel (E.P. 271,780, 10.2.27).—A roller grinding mill is provided with four or any greater even number of rolls so placed that any roll co-acts with two others, forming a number of grinding nips equal to the number of rolls. The rolls rotate successively in opposite directions, and at a speed increasing from the first to the last, the material progressing in the same order. Alternate rolls are provided with adjustable bearings, so that the grinding gaps may be varied two at a time. B. M. Venables.

Machines for crushing ores, stone, rock, etc. C. Roscoe (E.P. 271,799, 29.3.27).—A jaw-crusher in which the eccentric shaft and toggle motion is outside the fixed jaw, and the swing jaw at the opposite end of the frame, the motion being transmitted by rods outside the frame. B. M. Venables.

Stills and the like. W. E. English and J. R. Hannan (E.P. 271,567, 26.2.26).—In heaters for stills containing a number of elements each comprising a steam supply tube with open end within an outlet tube with closed end, each element projects through and is detachably secured to a tube plate, and is provided with a valve on both the supply and drain tubes, and with a test cock outside the tube plate. The renewal of faulty elements while working is facilitated.

B. M. Venables.

Centrifugal machine. Sharples Specialty Co. (E.P. 268,321, 10.3.27. Conv., 26.3.26).—The bowl of a super-centrifugal machine is provided with a ported head and detachable cap; for cleaning, it is only necessary to remove the latter, the former remaining undisturbed so that the very accurate balance of the rotor may be preserved. B. M. Venables.

Obtaining and storing gases under pressure. C. W. P. Heylandt (E.P. 271,692, 28.8.26).—The liquefied gases having low b.p. are evaporated in a high-pressure tank by a source of heat, and are led as water-free compressed gases into another pressure-resisting tank. The tank in which the liquid is stored and evaporated is coated on the interior and/or exterior with a heat-insulating material to facilitate the introduction and slow evaporation of the liquid. W. G. Carey.

Fire-extinguishing liquid. K. Kubierschky and W. Schultze (U.S.P. 1,633,383, 21.6.27. Appl., 16.3.26. Conv., 3.3.25).—The liquid consists of liquid sulphur dioxide mixed with carbon tetrachloride.

H. Royal-Dawson.

Treatment of liquids. C. Lourens, Assr. to N. V. Algem. Norit Maatschappij (U.S.P. 1,634,154, 28.6.27. Appl., 11.11.22. Conv., 14.11.21).—See E.P. 163,505; B., 1921, 456 A.

Centrifugal machines and processes. L. D. Jones and A. U. Ayres, Assrs. to Sharples Specialty Co. (U.S.P. 1,634,245—6, 28.6.27. Appl., [A], 13.4.27, [B], 1.5.24).—See E.P. 233,327; B., 1926, 343.

[Ash-cooling screens for] furnaces. Babcock & Wilcox, Ltd. From Deut. Babcock & Wilcox Dampfkesselwerke A.-G. (E.P. 264,097—8, 24.9.26).

Arrangement for the accurate control of cooling, especially in furnaces. E. Roucka (E.P. 265,546, 27.10.26. Conv., 3.2.26).

[Precast concrete blocks for] tunnel linings. S. Johannesson (E.P. 272,397, 20.12.26. Conv., 12.11.26).

Method and apparatus for spraying. E. G. Gresham (E.P. 247,170, 23.1.26).

[Connexions for spouts of] centrifugal separator installations. Aktiebolaget Separator (E.P. 269,883, 5.4.27. Conv., 24.4.26).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Improvement in Eschka's determination of sulphur [in coal]. H. ter Meulen (Chem. Weekblad, 1927, **24**, 205—206).—The sulphur contained in the volatile products, which escapes in the ordinary determination, may be arrested by passing the gases mixed with air over incandescent asbestos and then through potassium hydroxide. The results with this modification are from 3 to 5% higher than with the ordinary method. S. I. Levy.

Velocity of reaction between carbon dioxide and different species of carbonised fuels. J. A. Leffler (Jernkontorets Annaler, 1926, **110**, 165—208).—A study of the reaction $CO_2 + C = 2CO$ at 650—1150° with birch, red fir, spruce, and beech charcoals, and at 800—1400° with metallurgical coke.

Chemical Abstracts.

Steaming in continuous vertical retorts: theory and practice. N. H. Humphrys (Gas J., 1927, **178**, 370—371, 506—507, 635—637).—Advantages of steaming coal in gas retorts include the resulting diluting and conserving actions, as well as a greater regularity in the quantity and quality of gas produced, and the possibility of working to a closer margin above the declared calorific value. The quality of the coke and tar is improved, as are the working conditions. Comparative figures are given for the actual and calculated yields of therms and total gas (including its individual components) obtained by introducing varying quantities of steam. W. T. K. Braunholtz.

Experiments on complete gasification. K. Bunte (Gas- u. Wasserfach, 1927, **70**, 617—621).—A weight balance, a carbon balance, a water balance, and a heat balance have been drawn up for a producer in operation at the Weisenau gas works. In a three days' run, the producer being in operation for two periods of 5 and 6 hrs., respectively, each day, 4·7 tons of coal (moisture 2·4%, ash 5·7%, volatile matter 33·0%) were gasified, producing 5335 cub. m. of gas (0°, 760 mm.), of 3410 kg.-cal./cub. m. mean gross calorific value. The total thermal value of the gas amounted to 49·4% of that of the coal used. Carbonisation of the coal and subsequent conversion of the coke into water-gas, after allowing a proportion for heating the retorts, would give 108·2 cub. m. of "mixed gas" of 3510

kg.-cal./cub. m. The total thermal value of the gas would amount to 48·5% of that of the coal, but by this process there would be obtained in addition 6·6% of tar. A. B. Manning.

Record of the gases from coke ovens at Routchenkovo. Feodoroff (Chim. et Ind., 1927, **17**, 729—736).—The installation consists of 4 batteries of 35 ovens each, the normal charge for each being 8 tons of coal and the duration of the coking 36 hrs. The products of distillation of the coal are worked up for the recovery of ammonia, benzol, and tar. About 8800 cub. m. of gas are produced per hour, the yield being 347 cub. m. per ton of coal carbonised in the ovens. The distribution of the gas at various stages of the process is tabulated. H. M. Langton.

Permissible sulphur content of gas oils for the production of [carburetted] water-gas. G. A. Brender à Brandis (Het Gas, 1926, **46**, 430—434; Chem. Zentr., 1927, I, 1096—1097).—Large-scale experiments have shown that, using gas oils containing 0·27—2·82% S, the sulphur content of the purified carburetted water-gas ranged from 16·6 to 27·2 g./100 cub. m., or, after allowing for 12·3 g. from the coke, from 4·3 to 14·9 g./100 cub. m., whilst purified coal gas contains at least 40 g./100 cub. m. The crude gas contained 0·22—0·51% H_2S, or 0·04—0·33% after allowing for 0·18% from the coke itself, as compared with 0·8% in coal gas. The required maximum sulphur content for gas oils of 0·5% is, therefore, without justification, for even with oils containing several times that amount the increase in the hydrogen sulphide of the crude gas, and in the sulphur compounds, particularly carbon disulphide, of the purified gas, are inconsiderable compared with the amounts present in coal gas. A. B. Manning.

Hydrocarbons in gas from the low-temperature carbonisation of coal. F. E. Frey and W. P. Yant (Ind. Eng. Chem., 1927, **19**, 488—492).—The gas from the carbonisation of a non-coking Utah coal in a current of superheated steam at 700° has been analysed by fractional distillation (cf. B., 1927, 177), and a further study made of the hydrocarbon fractions. Acetylene and its homologues were absent. From the bromine addition products of the unsaturated constituents in the four-carbon-atom fraction *trans*-αβγδ-tetrabromobutane was isolated in amount equivalent to 0·002% of butadiene in the gas. Analysis of the dibromides showed that the butylene fraction (0·52% of the gas) consisted of 41% of *as*-dimethylethylene, 24% of ethylethylene, and 35% of *s*-dimethylethylene. The butane fraction (0·37% of the gas) consisted of 20% of *iso*butane and 80% of *n*-butane. Methyl*cyclo*propane was absent. A. B. Manning.

Separation of individual saturated and unsaturated hydrocarbons in coal gas by fractional distillation. F. E. Frey and W. P. Yant (Ind. Eng. Chem., 1927, **19**, 492—493; cf. preceding abstract).—A modification of the Shepherd and Porter method of gas analysis by fractional distillation at low temperatures is described, which permits the separation of the constituents into fractions containing (1) oxygen, nitrogen, hydrogen, carbon monoxide, and methane, (2) ethane and ethylene, (3) propane and propylene, and (4) butane and butylene, respectively. Each fraction can then be analysed in a simple Orsat apparatus. The method can be applied to 50—2000 c.c. or more of gas, according to its content of these hydrocarbons. An analysis requires 5—6 hrs. A. B. Manning.

Formation of carbides in the system metal–carbon–hydrogen and metal–carbon–oxygen. G. Meyer and F. E. C. Scheffer (Rec. trav. chim., 1927, **46**, 359—368).—Metal carbides rich in carbon, stable at low temperatures, and decomposed by heat, are probably formed during the production of synthetic petrol from water-gas in the presence of a metallic catalyst (Fischer and Tropsch, B., 1926, 475), and a nickel carbide of this character has, in fact, been isolated (Scheffer, Dokkum, and Al, A., 1927, 29; Meyer and Scheffer, *ibid.*, 220). The general principles involved in the study of the systems metal–carbon–hydrogen and metal–carbon–oxygen from the point of view of the phase rule are discussed. M. S. Burr.

Combustion limits under high pressure of mixtures of air with inflammable gases and vapours. E. Berl and G. Werner (Z. angew. Chem., 1927, **40**, 245—250).—The curve showing the lower limit of complete combustion of hydrogen–air mixtures starts at 7·0% of hydrogen at atmospheric pressure, rises steeply to 10·8% at 21 atm., then falls slowly to 8·4% at 210 atm., whereas the curve of the upper limit rises smoothly, concave to the pressure axis, from 60% of hydrogen at 1 atm. to 73·5% at 210 atm. with a weak ignition, but, with strong ignition, falls from 71·5% at 1 atm. to 69% at 10 atm., then slowly rises to 74% at 210 atm. The upper limit of complete combustion of hydrogen–air mixtures under reduced pressure falls fairly smoothly with decrease of pressure to 20% of hydrogen at 240 mm., whereas the lower limit remains constant down to 400 mm., then increases sharply to 20% at 240 mm., consequently air–hydrogen mixtures of any composition do not react with a weak ignition at pressures lower than 240 mm. The lower limit curves for ethylene–air and methane–air mixtures resemble in shape the corresponding hydrogen–air curve, both showing a high lower limit at 20 atm. The upper limit curve of ethylene–air mixtures starts at 16% of ethylene at 1 atm., and rises extraordinarily steeply to 68% at 90 atm., then very slowly to 71% at 380 atm. The combustion range of methane–air mixtures decreases from 6·6—12·7% of methane at 1 atm. to a minimum of 7·5—12% at 21 atm., then rapidly increases to 5·2—46% at 400 atm. Ignition of mixtures of ethylene or methane with insufficient air for complete combustion under just sufficient pressure for reaction to take place results in the preferential oxidation of the hydrogen, but with further increase in the pressure the proportion of carbon dioxide and especially of carbon monoxide formed increases rapidly; thus, of the oxygen in a 40% ethylene–air mixture, 83% appears as water, 3% as carbon dioxide, and 14% as carbon monoxide after ignition under 40 atm. pressure, whereas under 92 atm. these figures are 50, 10, and 40% respectively. Air saturated with ether vapour at 20° is outside the combustion zone at 1 atm., but

at 4 atm. the mixture commences to burn with a weak ignition, the rate of reaction reaching explosive violence at 19 atm., and finally falling to zero again at 30 atm. Similar results are obtained with benzene–air mixtures, and also by raising the temperature while keeping the pressure constant, there being an upper and lower limit of temperature for all pressures within which range only does combustion take place. A. R. Powell.

Conradson coke-test for motor oils. F. Schulz and M. Kohout (Petroleum, **23**, 554—557).—The Conradson coke-test as carried out by A.S.T.M. methods shows considerable experimental errors; these can be diminished if 2 g. of the oil are weighed into a porcelain crucible (Rosenthal 103, No. 4) and put into the cold Conradson apparatus, which is then heated for 20 min. with the Tirrill burner (height of flame 20 cm.), the bottom of the iron crucible being 5 cm. above the mouth of the burner. Results of such determinations are given.

W. N. Hoyte.

Effects of the corona discharge on petroleum. J. J. Jakosky (U.S. Bur. Mines, Tech. Paper, 1926, No. 375, 21 pp).—Treatment of cracked gasoline (approximately 36% unsaturated) with hydrogen activated by the corona discharge gave no perceptible effect. Natural gas on being submitted to the discharge at 20° and 100° slowly dissociated into carbon black and hydrogen; no polymerisation was noted; at 500° a very small amount of tarry matter, possibly due solely to the heat, was obtained. On submitting benzene vapour to the discharge a light, slightly viscous, brown liquid was slowly produced; the odour was similar to that of pinol. In experiments on vapour-phase cracking below 600° the effects of a high-frequency corona discharge and a low-frequency 60-cycle discharge were inappreciable. Above 600° the greatest cracking was obtained with the high-frequency discharge, and the least with no discharge. The unsaturation of the oil was least with no discharge, and greatest with the high frequency; thus at 800°, 54% unsaturation was observed with the high-frequency, 34% with the low-frequency; and 31% with no discharge. In general, more uncondensable gas was obtained when the high-frequency discharge was used. Tests on the precipitation of hot vapours by a corona discharge showed that below a certain critical temperature most of the vapour was precipitated on the walls of the tube; above this temperature no precipitation took place. The critical temperature coincided with the change in the vapour from a visible cloudiness to an invisible gas. The corona discharge itself produced no cracking of oil vapour. The increased cracking obtained by the use of the corona during the thermal cracking of oils appears to be due to precipitation of the hot oil vapours against the walls of the cracking tube, and to the decreased amount of channelling by the electric wind set up by the discharge.

W. N. Hoyte.

Regeneration of used lubricating and transformer oils. R. A. Wischin (Petroleum, 1927, **23**, 546—551).—The degradation of the oil is due to the atmospheric oxidation of the less stable parts of the oil, lactones or acids with carbonyl groups in particular being formed. Soaps of these acids are formed with alkali from the air or from entrained boiler-feed water. These soaps act as catalysts in furthering the oxidation, as also do the asphalts formed as oxidation products; the action thus takes place with increasing velocity. For oil regeneration the Benmann floridin process is recommended. The best working temperature is 60—90°, and the floridin must be granular. If necessary, the oil is subjected to a preliminary treatment (filtration or precipitation with sulphuric acid etc.) to remove metallic particles and carbon sludge. About 1 hr.'s contact is the optimum for all oils, and the size of granules should be chosen accordingly. The regenerated oil has a slight acidity due to lactones which are adsorbed with difficulty by floridin, but are not harmful in lubricating oil. W. N. Hoyte.

Helium in Canada. Elworthy.—See VII.

Use of alcohol in arson. Maulhardt.—See XX.

Patents.

Apparatus for separating coal from dirt and like foreign substances. W. H. Berrisford (E.P. 271,633, 10.5.26).—The jigger carries a step on to which material to be separated is pushed from a stationary step by virtue of the reciprocating motion of the jigger. As the jigger recedes below the stationary step, the material is pushed over the front edge of the movable step. Between these two steps may be situated an inclined plate, which divides the coal into two streams—clean coal and dirt—dust being removed by an air-current. R. A. A. Taylor.

Treatment of coal. Internat. Combustion Engineering Corp., Assees. of W. Runge and E. A. Packard (E.P. 264,169, 4.2.26. Conv., 12.3.25).—The coal is first pulverised in a mill in the presence of hot air or flue gases at about 100°, and is further heated by admixture with preheated air or flue gases in its passage to a separator. The degree of heating can be controlled and the coal either oxidised or carbonised as required. The separated material then falls into a retort which has at the bottom a coke-cooling chamber. The gases from the retort are used in the preheaters and may be admixed with air from the separator.

A. C. Monkhouse.

Apparatus for drying and distilling lignite, peat, non-coking coals, and other similar carbonaceous matter. H. Debauche (E.P. 270,921, 20.7.26).—The fuel is carbonised in a cylindrical firebrick retort which has in its centre a multi-ring cylinder consisting of a number of annular tapered collars. A variation in the thickness of the charge is obtained by varying the diameter of the collars, the carbonising space being 4 in. at the top of the retort and 2 in. wide at the bottom, according to the nature of the fuel. The distillation gases pass between the collars and are withdrawn by adjustable vertical pipes, arranged at different levels, to a common collecting main at the base of the retort. To prevent ingress of air, a suitable feeding hopper is used, and the semi-coke produced is withdrawn through a slide valve into a cooling box cooled by circulating water. After screening, the coke above 5 mm. is ready for sale and that below 5 mm. is further treated and briquetted. A. C. Monkhouse.

Transforming lignite and similar carbonaceous matter into fuel of high calorific value for industrial and domestic purposes. H. Debauche (E.P. 270,976, 29.11.26).—Semi-coke produced by methods described in, *e.g.*, E.P. 270,921 (cf. preceding abstract), is screened as it issues from the retort, the coke below 5 mm. size being treated in an enclosed system filled with an inert gas to prevent oxidation of the coke and to conserve the sensible heat for the subsequent manufacture of briquettes. The coke at 180° is elevated by an enclosed conveyor and fed on to a screen of mesh dependent on the grading required for the briquettes; the larger material passes to a crusher, and is then fed back to the screen. The coke is then fed by conveyor to a tower from which it is discharged to a worm conveyor, the opposite end of which is being fed with pitch from an adjacent tower. The proportions are controllable and the mixture is conveyed to the briquetting plant. In experiments with lignite, the semi-coke above 8 mm. was used for domestic grates, $5\frac{1}{2}$—8 mm. for boilers. The coke, 0—5 mm., gave a cohesion of 25% less than that of 0—1 mm., was 6% less dense, absorbed $4\frac{1}{2}$ times more water, and required 7·5% less pitch.

A. C. Monkhouse.

Manufacture of motor fuel. R. Cross, Assr. to Gasoline Products Co., Inc. (U.S.P. 1,631,401, 7.6.27. Appl., 12.9.25).—An anti-knock motor fuel is produced by extracting petroleum oil with liquid sulphur dioxide, separating the undissolved hydrocarbons, and cracking them.

C. O. Harvey.

Manufacture of gas suitable for heating and lighting purposes. S. J. B. Tully and O. E. Yeo (E.P. 271,765, 31.12.26).—The plant comprises a gas producer, a superposed retort, and a carburettor. Surrounding the retort is a chequer brickwork heating-jacket in communication with the producer and the carburettor, or the latter may constitute an independent portion of the heating jacket. The water-gas made is passed up through the retort, where it is enriched by coal gas, the mixture being led off independently of the carburettor; or the water-gas is passed through the heating jacket and then enriched by cracked oil vapours in the carburettor, whence it may pass to a gas-fixing or gas-treatment chamber.

R. A. A. Taylor.

Water-gas [from powdered fuel]. I. G. Farbenind. A.-G., Assees. of F. Winkler (G.P. 437,970, 28.9.22).—The air and the steam are blown into the producer in such a way that the whole of the charge of powdered fuel is given an up-and-down rotary motion.

A. B. Manning.

Recovering ammonia from gases. R. O. E. Davis and L. B. Olmstead (U.S.P. 1,631,720, 7.6.27. Appl., 13.6.21).—The ammonia is absorbed in a liquid cooled to about 0°, and is subsequently recovered by raising the temperature of the solution to 40—50°.

C. O. Harvey.

Distillation of solid carbonaceous material. M. J. Trumble (E.P. 271,740, 12.11.26).—The material is charged into a series of retorts, into the first of which superheated steam is introduced. The steam, now mixed with distillation products, is passed through the second retort, a part of the condensable distillation products being removed, and so on throughout the whole series. The steam and remaining distillation products are then passed through a superheater which restores the mixture to the initial temperature of the steam. The resulting hot mixture is then passed through a second series of retorts as through the first. The portion of the distillation products removed may be the heavier oils by condensation or other fractions by dephlegmation.

R. A. A. Taylor.

Distillation of oil shale. C. T. Loughrey (U.S.P. 1,631,637, 7.6.27. Appl., 5.5.21).—The shale is distilled by passage down a column up which a heating medium is passed, the vapours being withdrawn by the partial vacuum produced by injecting a liquid under pressure into the withdrawn vapours. The liquid undergoes re-circulation and injection.

C. O. Harvey.

Preparation of light hydrocarbons from heavy hydrocarbons or cyclic derivatives. A. J. Kling and J. M. F. D. Florentin (F.P. 607,155, 30.11.25. Conv., 12.6.25).—Heavy hydrocarbons are heated with hydrogen under pressure in the presence of a catalyst, *e.g.*, halogen compounds of magnesium, zinc, iron, aluminium, and chromium.

A. B. Manning.

Conversion of methane gas into hydrocarbons of higher carbon content. Pétrole Synthetique Soc. Anon., and A. Folliet (E.P. 271,767, 1.1.27. Addn. to E.P. 261,267; B., 1927, 100).—The prior process is modified by imposing upon the heated gases a vacuum of from ·20 to 50 cm. of mercury. The walls of the reaction chamber are of steel lined with refractory material.

C. O. Harvey.

Treatment of mineral oils by heat to obtain lower-boiling oils. W. J. Perelis (E.P. 271,601, 24.3.26).—In a cracking process, wherein the production of gas and coke is reduced and economy of heat is effected by means of interchangers, and which is suitable for the treatment of all types of oil (crudes, gas oils, kerosenes, etc.), the oil passes through a series of heating coils and is alternately heated at a cracking temperature, cooled during continued flow, and then reheated. The heaters are arranged for repeated reversals of the direction of flow.

C. O. Harvey.

Sweetening hydrocarbon oils. M. G. Paulus, Assr. to Standard Oil Co. (U.S.P. 1,628,423, 10.5.27. Appl., 28.5.25).—Hydrogen sulphide, contained in still gases, is dissolved in sour light hydrocarbon oils which are then treated with doctor (sodium plumbite) solution.

C. O. Harvey.

Preparation of emulsifying agents etc. I. G. Farbenind. A.-G., Assees. of W. Pungs and M. Luther (G.P. 438,180, 9.9.22).—The oxidation products of solid paraffins are treated with an alkali hydroxide in amount not more than necessary to saponify the saponifiable constituents. Hydrocarbons, or other liquids insoluble in water, and containing, if necessary, sulphur or other materials in solution, are added before and after saponification. In addition to its use as an emulsifying agent, *e.g.*, for drilling oils etc., the product may be used directly as a lubricating oil.

A. B. Manning.

Extraction of paraffin wax from brown coal. A. Mailhe (Addn. No. 30,634, 5.2.25, to F.P. 598,502;

B., 1926, 574).—The finely-powdered brown coal is treated with the vapours of organic solvents, and the extract so obtained with liquids in which paraffin wax is soluble, *e.g.*, the higher homologues of acetone.

A. B. MANNING.

Treatment [purification] of sludge acid. F. M. ROGERS, F. V. GRIMM, and G. L. WENDT, Assrs. to STANDARD OIL CO. (U.S.P. 1,630,074, 24.5.27. Appl., 14.8.24).—Waste sulphuric acid from oil-refining is diluted to *d* 1·38, and passed through a tank at 65°, in which it is subjected to electrolysis by a current of 3—3·5 amp./dm.^{2} to oxidise organic matter present.

T. S. WHEELER.

Rustproofing oil. R. E. WILKIN, Assr. to STANDARD OIL CO. (U.S.P. 1,630,101, 24.5.27. Appl., 13.3.25).—An oil for protecting ferrous metals against rust comprises a hydrocarbon oil heavier than gas oil, containing from 3—35% of sodium salts of sulphonic acids obtained in refining petroleum. T. S. WHEELER.

Coating gasometers. P. LECHLER CO., Assees. of R. MEZGER (U.S.P. 1,630,202, 24.5.27. Appl., 10.7.25. Conv., 11.9.22).—A solution of petroleum pitch in mineral oil is used to cover the surface of the water sealing a gasometer, so that with the movement of the bell, it coats the latter and protects it from corrosion.

T. S. WHEELER.

Carbonisation of fuel briquettes. E. B. A. ZWOYER, Assr. to GENERAL FUEL BRIQUETTE CORP. (U.S.P. 1,632,052, 14.6.27. Appl., 9.2.23).—See E.P. 211,101; B., 1924, 858.

Method and apparatus for centrifugally separating substances [*e.g.*, wax from oils]. L. D. JONES, Assr. to SHARPLES SPECIALTY CO. (U.S.P. 1,634,242—4, 28.6.27. Appl. [A], 9.4.27, [B, C], 7.10.21).—See E.P. 191,750; B., 1923, 703 A.

Emulsified solid grease. M. J. HEITMANN (U.S.P. 1,632,027, 14.6.27. Appl., 8.4.25. Conv., 10.4.24).—See E.P. 232,259; B., 1926, 478.

Production of petrol. F. TINKER (U.S.P. 1,632,011, 14.6.27. Appl., 25.9.22. Conv., 7.1.22).—See E.P. 195,465; B., 1923, 541 A.

Burner for use with liquid fuel, gas, etc. J. DAVIES (E.P. 271,575, 27.2.26).

Device [grate] for burning solid fuels. J. MARTIN (E.P. 265,191, 24.1.27. Conv., 29.1.26).

Coke-cooling plant. C. SCHWARTZ (E.P. 256,919, 5.1.26. Conv., 12.8.25. Addn. to E.P. 252,118; B., 1926, 780).

[Valves for] condensers for use in the recovery of by-products arising in the carbonisation of coal and the like. D. M. HENSHAW (E.P. 272,293, 11.3.26).

Cooling of coke and utilisation of the heat arising therefrom. COLLIN & CO., and J. SCHÄFER (E.P. 270,780, 14.1.26).

Apparatus for generating oil gas. A. E. HODGSON and N. C. JONES (E.P. 271,649, 31.5.26).

Handling coke discharged from coke ovens. N. V. SILICA EN OVENBOUW MIJ, and O. FRÖHLICH (E.P. 272,410, 19.1.27).

Carbonising plant (G.P. 436,515).—See III.

Activated charcoal (E.P. 257,917).—See VII.

III.—TAR AND TAR PRODUCTS.

Cresol-soap systems. JENČIČ.—See XII.

PATENTS.

Carbonising plant and generator for the production of low-temperature tar. C. ERHARD (G.P. 436,515, 13.12.24).—The gases and vapours evolved from a retort or producer pass through conduits, which are provided with blowers, into a dryer arranged vertically above the producer. In the dryer they pass through a heating arrangement which is supplied with a mixture of gas and steam from an external heater. The gases, steam, and other volatile products from the carbonisation of the fuel are withdrawn through a tar separator by means of a high-pressure blower, and compressed into a pressure chamber in which the steam condenses, whilst the gases pass to a holder. The gas and steam mixture supplied to both producer and dryer are taken from the circuit at a point between the tar separator and the high-pressure blower, and are heated in a special apparatus to such a temperature that the mixture of gases and vapours leave the dryer at 110°, the most favourable temperature for tar removal. A. B. MANNING.

Separation of liquids (E.P. 271,130).—See I.

IV.—DYESTUFFS AND INTERMEDIATES.

Behaviour of oxides of lead towards dyestuffs. II. A. N. ADAMSON and J. K. WOOD (J. Soc. Dyers and Col., 1927, 43, 192—194; cf. B., 1927, 324).—Hydrated lead monoxide shows no tendency to absorb basic dyes, neither dyeing of the oxide nor formation of precipitate taking place with Methylene Blue, Safranine T, Methyl Violet 10B, or Toluidine Blue. With acid dyes, such as Disulphine Green T and Patent Blue, the oxide is uniformly dyed without formation of precipitate, the absorption curves obtained in these cases by plotting the amount of dye remaining in solution against the amount taken up by the solid having more or less resemblance to ordinary adsorption isotherms. With Patent Blue it was found that a sample of oxide (moisture content 6·12%) prepared by precipitation with the minimum of alkali always adsorbed more dyestuff than a sample (moisture content 4·62%) obtained by precipitation with large excess of sodium hydroxide. Whilst the difference in adsorption powers may be partly due to the difference in moisture content, it may also be due to the presence of adsorbed alkali in the second sample, which would consequently be more electronegative than the first sample. With other acid dyes, *e.g.*, Methyl Orange, Orange II, Bordeaux B, and Orange IV, less regular results were obtained, dyeing of the oxide being accompanied by formation of a considerable amount of flocculent precipitate above the layer of hydrated oxide. Bordeaux B was removed in approximately constant amount independent of the concentration, and Orange IV was completely removed from dilute solutions by the sample of oxide having moisture content 6·12%. It is probable that absorption is accompanied by more or less chemical combination, and the compound obtained (probably the lead salt of the dye acid) itself absorbs a further amount of the dyestuff. The absorption curves in these four cases represent a resultant of the various processes.

R. BRIGHTMAN.

Patents.

Dyes and dyeing. B. Wylam, J. E. G. Harris, J. Thomas, and Scottish Dyes, Ltd. (E.P. 271,533, 21.1.26).—Sulphuric esters of leuco-compounds are obtained by adding the esterifying agent directly to the hyposulphite vat together with additional sodium hydroxide and a tertiary amine (pyridine). The esterifying agents used are alkyl chlorosulphonates, chlorosulphonic acid, oleum, sulphur trioxide, or the reaction product of any of these with pyridine. C. Hollins.

Dyes and dyeing. W. Smith, J. Thomas, and Scottish Dyes, Ltd. (E.P. 271,537, 26.1.26).—Flavanthrone and its derivatives (*e.g.*, dichloroflavanthrone) are reduced to leuco-compounds in cold concentrated sulphuric acid by treatment with a metal (copper). The leuco-compounds as prepared are stable, and may be used for conversion into leuco-esters by the processes of prior patents, and for dyeing and printing. C. Hollins.

Process of reducing vat dyes to their leuco-derivatives. C. H. Marschalk (E.P. 271,569, 26.2.26).—The dry vat dye is dissolved or suspended in an anhydrous organic solvent (particularly dimethylaniline) and is reduced with hydrogen in the presence of a suitable catalyst (*e.g.*, nickel, cobalt, iron, or copper on a pumice or kieselguhr carrier). The leuco-compound produced dissolves in the dimethylaniline, and is ready for conversion into a leuco-ester (" Indigo-sol "). C. Hollins.

Preparation of condensation products of the benzanthrone series. I. G. Farbenind. A.-G., Assees. of H. Neresheimer (G.P. 438,467, 26.10.24. Addn. to G.P. 407,838).—Purer products and better yields are obtained by using for the condensation of benzanthrones a diluent such as benzene, *o*-dichlorobenzene, etc. in the presence of a mixture of alkali hydroxide and alkali alkoxide as condensing agent. The reaction proceeds at low temperature, *e.g.*, 20—25° or 60°. All oxygen should be displaced by means of an inert gas. C. Hollins.

Manufacture of α-aroyl-β-naphthols and of condensation products of the benzanthrone series. I. G. Farbenind. A.-G., Assess. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 248,791, 9.3.26. Conv., 9.3.25).—β-Naphthyl esters of aromatic carboxylic acids are converted by acid condensing agents (aluminium chloride) in the presence of a diluent into α-aroyl-β-naphthols, which may be further condensed to give hydroxybenzanthrones or dihydroxydibenzpyrenequinones. In the absence of a diluent, sodium aluminium chloride brings about both reactions in one operation. 2 : 6-Dihydroxynaphthalene dibenzoate, m.p. 217°, heated with aluminium chloride in tetrachloroethane, yields 2 : 6-*dihydroxy*-1 : 5-*dibenzoylnaphthalene*, m.p. 275°, from which, by heating with sodium aluminium chloride at 140—150°, 1 : 6-*dihydroxy*-3 : 4 : 8 : 9-*dibenzpyrene*-5 : 10-*quinone*, together with 2 : 6-*dihydroxybenzanthrone*, m.p. 250—252°, are obtained. β-Naphthyl benzoate similarly yields 1-benzoyl-2-naphthol (phenyl 2-hydroxy-1-naphthyl ketone), m.p. 137—139°, and 4-hydroxybenzanthrone, m.p. 176°. C. Hollins.

Manufacture of vat dyes [of the dibenzanthrone series] containing nitrogen. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 254,742, 2.7.26. Conv., 2.7.25).—3-Bromo- or -chloro-benzanthrones are nitrated (probably in the 8-position), the products are reduced, and then fused with alkali, preferably with the addition of alcohol, to give vat dyes which give reddish-grey to black shades, becoming green when chlorinated. Dyes are thus obtained from 3-bromo-8(?)-aminoanthraquinone, m.p. 274—278°, prepared from 3-bromonitroanthraquinone, m.p. 290°, and 3-chloro-8(?)-aminoanthraquinone, m.p. 290—291°, from 3-chloronitroanthraquinone, m.p. 284—285°. C. Hollins.

Manufacture of halogenated indanthrones. Brit. Dyestuffs Corp., Ltd., W. D. Rogers, W. V. Stubbings, and F. W. Emerson (E.P. 271,181, 1.3.26).—The halogenation of indanthrone purified by the sulphuric acid method of E.P. 9269 of 1914 (B., 1915, 900) yields products fast to chlorine. The degree of halogenation necessary is much lower than when a purified indanthrone is used. C. Hollins.

Manufacture of carbocyclic or heterocyclic compounds. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 251,997, 5.5.26. Conv., 5.5.25).—Fluorosulphonic acid is used for ring-closure in the manufacture of carbocyclic and heterocyclic compounds. Improved yields are claimed, due to smaller tendency to sulphonation. Examples of the cyclisation of thioglycollic acids to thioindoxyls and thioindigos (fluorosulphonic acid being also an oxidising agent), and of 4-amino-1-*o*-carboxyanilinoanthraquinone to the acridone described in E.P. 25,184 of 1910 (B., 1911, 1111) are given. C. Hollins.

Triarylmethane dyes from tetra-alkyldiaminobenzophenone and arylated ethylenediamines. Triarylmethane dyes from tetra-alkyldiaminobenzophenones and di-α-naphthylethylenediamine. I. G. Farbenind. A.-G., Assees. of H. Polikier and H. Hähle (U.S.P. 1,629,884—5, 24.5.27. Appl., [A, B], 6.5.26. Conv., [A], 14.3.25; [B], 9.4.25).—(A) *s*-Diphenylethylenediamines non-substituted in the 4 : 4′-positions, and (B) *s*-di-α-naphthylethylenediamine, are condensed with (A) 1 or (B) 2 mols. of a 4 : 4′-tetra-alkyldiaminobenzophenone, *e.g.*, 4 : 4′-tetramethyldiaminobenzophenone, by heating with phosphoryl chloride in presence of toluene at 100°, to yield *dyes*, which give strong, clear, fast, violet shades on tannin-mordanted cotton and wool. T. S. Wheeler.

Manufacture of anthraquinone derivatives. British Dyestuffs Corp., Ltd., and A. Shepherdson (E.P. 271,602, 27.3.26).—In the condensation of unsulphonated arylamines with unsulphonated halogenoanthraquinones the large excess of arylamine usually employed as a diluent is replaced by sodium acetate crystals or sodium acetate and water. The quantity of arylamine may be reduced to about 1·5 mols. instead of 6—10 mols. C. Hollins.

Manufacture of azo dyes. I. G. Farbenind. A.-G., Assees. of A.-G. für Anilin-Fabr. (E.P. 253,488, 16.4.26. Conv., 9.6.25).—Diazo compounds are coupled with a variety of azines obtained by alkaline fusion of naphthaphenazines and dinaphthazines containing a sulphonic group in position 8 in the naphthalene ring and also

containing other hydroxyl or sulphonic groups. The azines have affinity for cotton and may be coupled on the fibre. The alkaline fusion product of the azine prepared by condensing 1 : 2-naphthaquinone-4 : 8-disulphonic acid or 4-hydroxy-1 : 2-naphthaquinone-8-sulphonic acid with *o*-phenylenediamine, is coupled with diazotised sulphanilic acid for a yellow wool dye; with diazotised naphthionic acid for a red wool dye; with diazotised 4-aminoazobenzene-4′-sulphonic acid for a red wool or direct dye; with diazotised *p*-toluidine-*m*-sulphonic acid for lake browns; with diazotised 4-chloro-2-aminophenol-5-sulphonic acid or 6-nitro-2-aminophenol-4-sulphonic acid for a chrome violet-brown; on the fibre with diazotised *p*-nitroaniline (yellow) or tetrazotised dianisidine (violet-brown). Other azines used are obtained by alkaline fusion of the condensation products of 4-hydroxy-1 : 2-naphthaquinone-8-sulphonic acid with 1 : 2-naphthylenediamine, or 4-nitro-*o*-phenylenediamine; or of 1 : 2-naphthaquinone-3 : 8-disulphonic acid with *o*-phenylenediamine; or of 1 : 2-naphthaquinone-8-sulphonic acid with 1 : 2-naphthylenediamine-6-sulphonic acid. C. Hollins.

Water-insoluble colours or dyes. E. C. R. Marks. From E. I. du Pont de Nemours & Co. (E.P. 271,580, 2.3.26).—Dyes for nitrocellulose products and for spirit varnishes are obtained by converting acid dyes into their aryl-, diaryl-, or triaryl-guanidine salts, which are insoluble in water but soluble in organic solvents. Thus, di-*m*-xylylguanidine, dissolved in acetic acid, is added to an aqueous solution of Chrysophenine, giving a yellow precipitate soluble in acetone, methyl alcohol, or ethyl acetate. Di-*o*-tolylguanidine, diphenylguanidine, and phenyldi-*o*-tolylguanidine may also be used, and the process may be applied to all dyes containing sulphonic or carboxylic acid groups. C. Hollins.

Manufacture of intermediate products for azo dyes. Brit. Synthetics Ltd., and E. B. Higgins (E.P. 271,146, 17.2.26. Addn. to E.P. 262,958; B., 1927, 102).—The processes of the earlier patent are extended to the preparation of 2 : 3-hydroxynaphthoic arylamides from arylamines containing negative substituents, *e.g.*, *m*- and *p*-nitroanilines, *m*-nitro-*p*-toluidine, 2 : 5-dichloroaniline, sulphanilic acid. C. Hollins.

Manufacture of diacylacetylarylenediamine. A. Zitscher and R. Schmitt, Assrs. to I. G. Farbenind. A.-G. (U.S.P. 1,634,090, 28.6.27. Appl., 15.5.23).—See E.P. 211,772; B., 1924, 413.

Production of *N*-dihydro-1 : 2 : 1′ : 2′-anthraquinoneazine. E. C. R. Marks. From Du Pont de Nemours & Co. (E.P. 271,942, 2.3.25).—See U.S.P. 1,580,700; B., 1926, 577.

Manufacture of chromable azo dyestuffs. F. Straub and H. Schneider, Assrs. to Soc. Chem. Ind. in Basle (U.S.P. 1,633,874, 28.6.27. Appl., 21.7.26. Conv., 24.9.25).—See E.P. 258,854; B., 1927, 518.

Manufacture of azo dyestuffs and chromium derivatives thereof. Soc. Chem. Ind. in Basle (Ges. für Chem. Ind. in Basel) (E.P. 254,708, 25.6.26. Conv., 3.7.25).—See U.S.P. 1,623,005; B., 1927, 469.

Coloured leather (E.P. 248,767).—See XV.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Pancreatic digestion of wool. L. Meunier, P. Chambard, and H. Comte (Compt. rend., 1927, **184**, 1208—1210).—Wool which has been allowed to swell in alkaline solutions is attacked by pancreatic enzymes. With a concentration of enzyme of 2 g./litre, the binding cement between the cells is broken down. When the alkaline solution has p_H 10, the cells enclosed in the external sheath are detached, whereas when the concentration of the enzyme is increased to 5 g./litre, the sheath itself is broken down. There is no pancreatic attack when wool is steeped in a solution having p_H 4 (the isoelectric point of the wool). M. Clark.

Structural picture of the cotton hair as an aid to the interpretation of some phenomena. F. P. Slater (J. Soc. Dyers and Col., 1927, **43**, 181—185).—From consideration of the growth of the cotton hair and from examination of the mature hair in elevation and in cross-section, a structural picture of the cotton hair is developed which shows that there is an enormous surface on which dyes can be adsorbed. The variation in shade of dyed yarns with the type of cotton used, and the fact that the depth of shade corresponds with the order of wall thickness are in accordance with the structural picture thus developed. The large variation in wall thickness with any pure strain sample of cotton accounts for variations in the characteristics of raw cotton. Immature hairs in which no secondary wall cellulose has been deposited are different, both chemically and physically, from normal hairs, and often emerge undyed from the dyebath. R. Brightman.

Determination of copper number [of cellulose]. D. Clibbens and A. Geake (Papier-Fabr., 1927, **25**, 401).—The superiority of the Schwalbe–Braidy over the Schwalbe–Hägglund method in the range of low copper numbers is well established, but it may be that for higher copper numbers and for the investigation of wood cellulose the former method is unsuitable, without modification, and that the latter is better. This suggests that the copper number is dependent on the physical or mechanical structure of the cellulose, and it is doubtful, therefore, whether a single method is suitable for both cotton and wood cellulose. The degree of swelling of cotton cellulose exerts no influence on its Methylene Blue absorption, *e.g.*, mercerised cotton has no higher absorption than unmercerised. If, however, cotton is dried after treatment with dilute sulphuric or phosphoric acid, an increased absorption is given which is dependent, not upon the swelling of the cellulose, but upon the fixation of acid. Cottons of different origins show markedly different absorptions, possibly due to purely chemical differences, and not to different degrees of swelling. The absorption of water, which, in a physico-chemical sense, is taken as the most exact measure of the degree of swelling, shows no significant differences for cottons of different origin. For textile purposes both copper number and Methylene Blue absorption measurements are necessary if the conditions of oxidation are unknown, since the production of oxycellulose may be characterised either by low Methylene Blue absorption and high copper number or by high absorption and low copper number, according to the p_H value of the bleaching liquor. B. P. Ridge.

PATENTS.

Method of chemically varying artificial fibres. HEBERLEIN & Co., A.-G. (E.P. 261,794, 22.11.26. Conv., 21.11.25).—Artificial silk fibres are treated with a halogen compound of phosphorus in a suitable solvent (*e.g.*, phosphorus trichloride in toluene) in the absence of alkali. In order to neutralise acid liberated, an alkaline salt (sodium carbonate) or an agent which neutralises acid without having an alkaline reaction (sodium acetate) is added, if required. Viscose silk so treated is rendered insensitive to substantive dyes, whilst acetate silk may afterwards be dyed with basic dyes. B. P. RIDGE.

Treating crape waste. G. BONNARD (E.P. 270,727, 4.5.27. Conv., 10.5.26).—The material is passed through an opening machine, carded, the sliver cut up into lengths of about 4 cm. (which are re-carded if necessary), treated with boiling water containing soap in order to separate the fibres, washed, dried, and again carded before spinning. B. P. RIDGE.

Manufacture of artificial silk. SYNTHETA, A.-G. (E.P. 265,577, 27.1.27. Conv., 4.2.26).—The threads (of nitrocellulose or acetate silk) are spun directly into an atmosphere of the hot solvent vapours with the exclusion of any other atmosphere, the desired concentration of this atmosphere being maintained by the removal and condensation of surplus vapour. The difficulty of recovering solvent from dilute mixtures with air is thus avoided. B. P. RIDGE.

Manufacture of artificial silk. D. A. McKENZIE, Assr. to VISCOSE Co. (U.S.P. 1,630,197, 24.5.27. Appl., 28.12.23).—The annular fibre cake of fragile artificial silk formed in the spinning box is placed on a carrier which is then rotated. Washing water is delivered to the interior of the cake, and passes through it under the action of centrifugal force. The washed material is transferred on the carrier to the drying chambers and thence to the skeining machines. T. S. WHEELER.

Production of carbohydrate compounds. W. HARRISON (E.P. 264,261, 24.10.25).—Cellulose or starch xanthate, prepared in the usual manner, is oxidised with nitrous acid in the presence of acetic acid and in the absence of other mineral acid. The bulky, spongy mass of sulphide $(RO\cdot CS)_2S$, or disulphide, $(RO\cdot CS\cdot S)_2$, so formed is broken up and washed with water. In place of nitrous acid, nitrogen trioxide or tetroxide may be used, and the process is applicable to other carbohydrates or carbohydrate mixtures. C. HOLLINS.

Extraction of pure cellulose from the bagasse of sugar cane. E. C. H. VALET (U.S.P. 1,630,147, 24.5.27. Appl., 9.8.26. Conv., 14.6.26).—Bagasse, cut into small portions, is extracted with 3—5% calcium oxide solution, and is then heated under 1·5 atm. pressure with a mixture of 6% sodium hydroxide solution and 2—3% sodium sulphite solution for 4—6 hrs. The cellulose obtained is cleaned by treatment with steam and bleached. T. S. WHEELER.

Production of plastic masses from cellulose derivatives. PATHÉ CINÉMA (ANC. ÉTABL. PATHÉ FRÈRES) (F.P. 606,969, 10.3.25).—Non-inflammable products are obtained by dissolving cellulose derivatives, such as cellulose acetate, nitrate, or nitroacetate, or ethyl- or benzyl-cellulose, in phosphoric acid esters containing one or more aliphatic radicals and one or more aromatic radicals, such as diphenylmethyl, diphenylethyl, phenyldimethyl, and phenyldiethyl phosphate. L. A. COLES.

Production of celluloid-like masses. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 247,174 and 263,076, 27.1.26. Conv., [A], 9.2.25; [B], 21.12.25. Addn. to E.P. 247,174, preceding).—(A) Phenol esters of phosphoric acid, of the highest purity, are mixed with nitrocellulose containing not more than 11% N, with or without the addition of solvents and diluents, and the mixture is worked up in the usual manner. (B) Softening agents (*e.g.*, esters of phthalic acid etc.) or hardening agents (*e.g.*, chloral etc.) are added prior to or during working up. B. P. RIDGE.

Manufacture of paper pulp. Treatment of plant material. F. K. FISH, JUN. (U.S.P. 1,633,730—1, 28.6.27. Appl., 11.7.25. Conv., [A], 22.12.24, [B], 20.12.24).—See E.P. 244,788—9; B., 1926, 871.

Preparation of paper pulp. F. K. FISH, JUN. (U.S.P. 1,633,736, 28.6.27. Appl., 24.5.26. Conv., 7.7.25).—See E.P. 255,030; B., 1927, 474.

Means for extracting fibres from the stalks of flax, hemp, and like plants. B. P. VON EHRENTHAL and K. SCHOLZ (E.P. 271,938, 10.12.25).

Continuously operating machine for phosphating and washing fabrics. M. RATIGNIER (E.P. 272,073, 13.9.26).

Vulcanised products (E.P. 271,553).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Testing dyestuffs for fastness to washing. S. R. TROTMAN (J. Soc. Dyers and Col., 1927, **43**, 192).—A dyeing is made under standard conditions, and, after washing and drying, a weighed portion of the dyed sample is tested for fastness to washing under the standard conditions. At the end of the test the soap liquor is squeezed back into the bath, the sample washed with hot water, and the washings are added to the bath. The soap liquor and washings are cooled and made up to a measured volume. The quantity of dyestuff in this solution is then determined by placing an aliquot part in a Nessler cylinder and running a standard solution of the dyestuff, *e.g.*, 0·01%, into a second cylinder containing the same volume and concentration of soap solution as the diluted dye liquor, until the colours of two solutions match. More accurate results can be obtained with a Lovibond tintometer. From the volume of dye solution used the quantity of dyestuff extracted during washing is calculated and expressed as a percentage on the weight of fabric used. The method affords consistent numerical results on the same dyestuff, and enables a number of dyestuffs to be arranged in order of fastness to washing. R. BRIGHTMAN.

PATENTS.

Grounding or padding of textile materials. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (E.P. 271,543, 16.2.26).—Grounding and padding baths are improved by the addition of alkylated and/or aralkylated aromatic, or partly hydrogenated aromatic, sulphonic acids or sulphonates, phenolic derivatives being excluded.

Examples are: sulphonated benzyl- or polybenzylnaphthalenes; condensation products of naphthalenesulphonic acids with alcohols or mixtures of alcohols or with benzyl chloride, or tetralin dichlorides, or chlorinated paraffin oil; tetralinsulphonic acid condensed with *cyclo*hexanol; naphthalene condensed with benzyl chloride and butyl alcohol and sulphonated; anthracene benzylated and sulphonated. The effect of these substances is to assist penetration of the fibre and also to disperse the grounding or padding agent when this is not completely soluble. C. HOLLINS.

Production of azo dyes on silk. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 253,865, 18.3.26. Conv., 20.6.25).—Degummed silk is impregnated at 20—30°, preferably in presence of a protective colloid, with a non-sulphonated arylamide of 2:3-hydroxynaphthoic acid or with a coupling component containing a reactive methylene group, and then coupled with a diazo component. Raw silk may also be degummed and bleached after dyeing in this manner. 2 : 3-Hydroxynaphthoic β-naphthylamide coupled with diazotised 4-nitro-*o*-anisidine gives a scarlet; bisacetoacetyltolidine with diazotised *o*-chloroaniline a yellow which does not bleed during degumming; bis-2 : 3-hydroxynaphthoyldianisidine with diazotised 5-nitro-*o*-anisidine a bordeaux. C. HOLLINS.

Production of fast mixed dyeings on silk. I. G. FARBENIND. A.-G. (E.P. 263,088, 21.7.26. Conv., 21.12.25. Addn. to E.P. 253,865; preceding abstract).—The raw or degummed silk is impregnated with a coupling component as in the prior patent and with the leuco-compound of a vat dye. The leuco-compound is oxidised and the ice-colour developed with a diazo component. Thus silk, impregnated with leuco-dibenzamidoanthrarufin and 2 : 3-hydroxynaphthoic *p*-anisidide, oxidised and developed with diazotised 5-nitro-*o*-anisidine, is dyed bordeaux; bisacetoacetyltolidine and leuco-Indanthrene Blue-Green B, oxidised and developed with *o*-chloroaniline, give a green. C. HOLLINS.

Preparations for dyeing cellulose esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 271,550, 22.2.26).—Insoluble or sparingly soluble compounds, suitable for dyeing cellulose esters either directly or by coupling on the fibre, are mixed with molasses. Other substances (protective colloids) may be added. Vat dyes are excluded. A preparation of aminoazobenzene ground with about 3 pts. of molasses and a little water gives on mere dilution a dyebath suitable for dyeing cellulose acetate. An intimate mixture of 1 : 4 : 5 : 8-tetra-aminoanthraquinone, molasses, sulphite-cellulose waste liquor, and sodium di-*p*-tolyl phosphate forms a homogeneous paste from which no dye separates on dilution. C. HOLLINS.

Dyeing, printing, or stencilling of cellulose acetate materials. R. CLAVEL (E.P. 270,987, 10.11.25). —Cellulose acetate materials after being weighted with tin phosphate or other insoluble salts (particularly of tin, zinc, tungsten, or aluminium) have affinity for direct, acid, basic, and other dyes, *e.g.*, Janus Black D, Rhodamine G, Capri Blue, etc. C. HOLLINS.

Cross-dyeing cellulose fabric. J. C. WATSON, Assr. to T. P. GATES (U.S.P. 1,629,769 and 1,629,770, 24.5.27. Appl., 20.7.26).—(A) A process for cross-dyeing woven cotton and artificial silk fabrics comprises treating the warp threads during slashing (sizing) with a naphthol or other azo-dye coupling component, treating the filling threads with a different naphthol, weaving the fabric, and then treating it with a diazotised aromatic amine. Alternatively, the warp threads alone are treated. (B) The warp and filling threads are pretreated with leuco-compounds of two different vat dyes, and the woven fabric is treated with an oxidising agent. T. S. WHEELER.

Dyeing films, plates, and other non-fibrous articles constructed of cellulose. E. CZAPEK (F.P. 605,661, 3.11.25).—Solutions of dyestuffs soluble in alkalis, but insoluble, or nearly so, in acids, are added to alkaline cellulose solutions, especially viscose solutions, and the cellulose and dyestuff are precipitated simultaneously with sulphuric acid or with ammonium chloride and hydrochloric acid. L. A. COLES.

Washing or dyeing machine. A. K. CROAD. From COWLES ENGINEERING CORP. (E.P. 271,749, 27.11.26).

Dyes and dyeing (E.P. 271,533 and 271,537).—See IV

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

U.S. Government master specification for soda ash. (U.S. Bur. Standards, Circ. No. 314, Nov. 23, 1926. 5 pp.).—The loss in weight after drying a sample for 1 hr. at 150—155° must not exceed 1%, and the total alkalinity of the material thus dried must not be less than 58%, calculated as Na_2O, matter insoluble in water shall not exceed 0·25%, and neither hydroxide nor bicarbonate must be present. 30 g. shall have a volume of 55—65 c.c., and not more than 0·5% shall fail to pass a No. 4 sieve (mesh 0·187 in.). Hydroxide is tested for by adding 10% barium chloride solution to a solution of the soda ash until no more precipitate forms, filtering, and adding a drop of 10% silver nitrate solution to the filtrate. A dark coloration indicates hydroxide. For the total alkalinity the solution is titrated with 0·5*N*-sulphuric acid, using methyl orange; another similar amount is then taken and about 1 c.c. less acid is added and the free carbon dioxide expelled by boiling, after which methyl orange is added and the titration completed. For bicarbonate the original solution is titrated with 0·5*N*-caustic soda until a drop produces instantly a dark colour with silver nitrate on a spot plate. W. G. CAREY.

U.S. Government master specification for caustic soda (lye, for cleaning purposes). (U.S. Bur. Standards, Circ. No. 315, Nov. 23, 1926. 3 pp.).—Not less than 90% of sodium hydroxide and not more than 4% of carbonate calculated as sodium carbonate must be present. For the hydroxide determination an aqueous solution of 20 g./litre is made, 50 c.c. of 10% barium chloride solution are added to 50 c.c. of this solution and titrated with 0·5*N*-sulphuric acid, using phenolphthalein as indicator; the carbonate is determined in the same solution by adding methyl orange and titrating the barium carbonate. W. G. CAREY.

U.S. Government master specification for laundry soda (washing soda). (U.S. Bur. Standards,

Circ. No. 316, Nov. 23, 1926. 4 pp.).—Laundry soda is a mixture of sodium carbonate and bicarbonate, the total alkalinity of which shall be 39—41%, calculated as Na_2O, and matter insoluble in water must not exceed 0·1%. If the sample contains hydroxide it is rejected. Determinations are as for soda ash (cf. *supra*).

W. G. CAREY.

U.S. Government master specification for granular sodium carbonate (monohydrate crystals). (U.S. Bur. Standards, Circ. No. 317, Nov. 23, 1926. 5 pp.).—The total alkalinity of this substance, calculated as Na_2O, must not be less than 48·5%, matter insoluble in water must not exceed 0·1%, and neither hydroxide nor bicarbonate must be present. The technique of the hydroxide test and determination of total alkalinity and bicarbonate are the same as for soda ash (cf. *supra*). W. G. CAREY.

Decomposition of phosphate rock by sulphuric acid. W. STOLLENWERK (Z. angew. Chem., 1927, **40**, 613—620).—Phosphate was ground to a paste with water, the paste added to sulphuric acid, the mixture stirred for a given time, then filtered, and the residue washed free from acidity. Curves and tables were obtained showing the relation between the percentage of phosphoric acid dissolved and time, temperature, and concentration of the sulphuric acid used. The conversion is decreased, though the commencement of the reaction is hastened, by rise in temperature; the use of stronger sulphuric acid retards the reaction, though at lower temperatures (18°) the conversion is not decreased. Rise of temperature also increases the proportion of iron and aluminium dissolved. This quantity also varies with the time, but is not much affected by the strength of the sulphuric acid used. The behaviour of the reaction mass suggests that the decomposition of calcium phosphate by sulphuric acid proceeds in two stages. To test this, the sulphate content of the filtrate was determined. After allowing for the solubility of calcium sulphate in phosphoric acid, which was redetermined, a large deficiency was found in the free sulphuric acid content as deduced from the phosphoric acid liberated. It was shown in an indirect manner that solid calcium hydrogen sulphate is not formed under these conditions. Analyses of the filtrates suggested the following reactions (1) in the cold, $3Ca_3(PO_4)_2 + 9H_2SO_4 = 2CaHPO_4 + 4H_3PO_4 + 7CaSO_4 + 2H_2SO_4$; (2) at 50°, $3Ca_3(PO_4)_2 + 9H_2SO_4 = Ca_3(PO_4)_2 + 4H_3PO_4 + 6CaSO_4 + 3H_2SO_4$. Probably the dilution of the sulphuric acid checks the reaction, but the gradual conversion of anhydrite into gypsum effects a reconcentration. A microscopical examination confirmed this view. The complete reaction $Ca_3(PO_4)_2 + 3H_2SO_4 = 3CaSO_4 + 2H_3PO_4$ may be taken to be unimolecular as tricalcium phosphate is practically insoluble. A calculation of the velocity coefficient on this assumption gave the values 0·0159 at 18° and 0·0263 at 30°. The constancy of these justifies the assumption, but at higher temperatures the reaction appears to be bimolecular.

C. IRWIN.

Volumetric assay of iodides. A. J. JONES (Pharm. J., 1927, **118**, 723—724).—Iodides in presence of chlorides and bromides are determined by titration with standard potassium iodate solution in presence of hydrochloric acid. E. H. SHARPLES.

Helium in Canada. R. T. ELWORTHY (Canada Dept. Mines, 1926, No. 679. 64 pp.).—Technical research in the extraction of helium from natural gases in Canada and the United States has shown the existence of several rich helium-bearing supplies, and the sources of helium have been studied, with the result that more than 25 million cub. ft. have been extracted from natural gas in Texas. The methods used for extraction depend on the removal of gaseous helium (b.p. — 269°) from the other liquefied constituents of the gas. The procedure of analysis of gases containing helium is described. From the results of large-scale experimental plants operated by the Linde Air Products Corp., Air Reduction Co., and the Jeffreys–Norton process, it was decided to instal a Linde plant to treat 5 million cub. ft. of gas per day, and the cost of extraction has been reduced progressively to $24 per 1000 cub. ft. The helium content of gases at two points in Alberta and four points in Ontario ranges from 2 to 8 pts. per 1000, compared with 0·01—2 recorded for samples of natural gas from other parts of the world, and from the six positions a supply of over 5 million cub. ft. of helium per year is estimated, whilst the U.S. authorities state that it would be possible to extract yearly 50 million cub. ft. from gas which is being regularly used. The advantages of helium as compared with hydrogen for use in airships, apart from non-inflammability, are the lower diffusion of helium and the possibility of heating the gas-bags to expand the gas and counteract its greater density. Helium contaminated by diffusion of air into the gas-bags is purified by liquefying the contained oxygen and nitrogen or by the absorption of these gases by means of activated charcoal at low temperatures. The cost of storage and transportation of helium has been materially reduced by the development of large high-pressure steel cylinders. Cylinders 40 ft. long, $4\frac{3}{4}$ ft. in diameter, with walls 2 in. thick, charged to 2000 lb./sq. in. have a capacity of 200,000 cub. ft. of gas. C. A. KING.

Formation of carbides. MEYER and SCHEFFER.—See II.

Iron in silicates. SARVER.—See VIII.

PATENTS.

Production of sulphuric acid. METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 262,397, 22.10.26. Conv., 7.12.25).—A mechanical mixing apparatus producing intimate contact between gases containing sulphur dioxide and finely-divided nitrosylsulphuric acid is employed in addition to a Glover tower plant of the usual type, and a similar mixing apparatus is interposed between each pair of chambers or towers (cf. E.P. 184,966; B., 1922, 858 A). W. G. CAREY.

Manufacture of sulphuric acid. E. L. LARISON (U.S.P. 1,631,139, 7.6.27. Appl., 3.3.26).—Acid denitrated in a denitrating tower is delivered to the inlet of a nitrogen oxide recovery tower, and a portion of the acid from this tower is returned to the inlet, the remainder being sent to the denitrating tower. W. G. CAREY.

Stabilising hydrocyanic acid. DEUTSCHE GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESSLER, and F. KERSCHBAUM (E.P. 271,236, 5.5.26).—Kieselguhr for

absorbing and stabilising hydrocyanic acid is roasted, preferably in presence of a large amount of air, and is then freed from basic substances by treatment with acids or acid salts, the excess of acid being removed by washing. Esters which act as irritants, *e.g.*, chloroacetates, or which have a stabilising effect, *e.g.*, methyl chloroformate, may be used instead of acids.

W. G. CAREY.

Production of phosphoric acid. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 246,128, 5.1.26. Conv., 17.1.25).—In the combustion of phosphorus (or of gases containing it) with air, corrosion of the wall of the combustion chamber is avoided by continuously wetting the wall with phosphoric acid solution, either by employing a horizontal rotating tube as combustion chamber or, if an empty vertical shaft is used, by irrigating the walls with the solution. W. G. CAREY.

Manufacture of phosphorus or phosphoric acid and concurrently of activated charcoal. SOC. POUR L'EXPLOIT. DES PROC. E. URBAIN (E.P. 257,917, 24.8.26. Conv., 4.9.25).—Monocalcium phosphate or phosphoric acid is heated with considerable excess of charcoal or carbonisable organic matter for 8—12 hrs., the mass being in granular form. The phosphorus is collected and purified, or is burnt before condensation, the activated charcoal which remains being washed with acid to dissolve any basic phosphate.

W. G. CAREY.

Discharging ammonium sulphate from saturating tanks. F. J. COLLIN, A.-G. ZUR VERWERTUNG VON BRENNSTOFFEN U. METALLEN (E.P. 268,309, 21.2.27. Conv., 24.3.26).—Compressed air is employed, and is heated in the saturating tank by the gases and vapours, thus cooling the vapours so that condensable substances are separated therefrom. W. G. CAREY.

Purification of gases for the synthesis of ammonia. K. MÜLLER (E.P. 261,388, 10.11.26. Conv., 12.11.25).—After preliminary cleaning with lime or caustic alkali the gases are freed from final traces of oxygen, carbon monoxide and dioxide, sulphurous gases, and steam by absorbing the impurities in sodium hydride under pressure at 200° or higher. The active surface of the hydride is increased by mixing therewith inorganic substances, especially metals. W. G. CAREY.

Treatment of zinc chloride-bearing solutions. ORKLA GRUBE-AKTIEBOLAG (E.P. 252,388, 21.5.26. Conv., 22.5.25).—The zinc is precipitated with less than the equivalent amount of lime at 30—40°, and, after removal of the mother-liquor, the resulting basic zinc chloride is treated with not more than an equivalent quantity of lime at about 70° to form zinc oxide, which is dried and sintered at 1000—1200° without removal of the calcium chloride. The quantity of lime is adjusted so that the subsequent sinterings remove cadmium etc. by volatilisation of the chloride. W. G. CAREY.

Utilising the liquors derived from chloridising roasting pyrites cinder and the like. REYMERSHOLMS GAMLA IND. AKTIEBOLAG (E.P. 260,294 and 260,297, [A], 22.10.26; [B], 23.10.26. Conv. [A, B], 24.10.25).—(A) The liquor first obtained on lixiviating the roasted material and containing salts mostly in the form of chlorides is kept separate from the later leachings, which consist mostly of sulphates. Copper is precipitated from the chloride liquor by means of zinc, and the solution free from iron is suitably treated for the recovery of silver and cobalt, after which it is cooled to crystallise sodium sulphate. The sulphate liquor is treated with cement copper to precipitate cuprous chloride, and, after treatment with an alkaline reagent to precipitate other metals, the liquor is evaporated to produce sodium sulphate. (B) Sufficient chlorine ions are allowed in the sulphate liquor to be equivalent to the cupric copper ions, and the cuprous chloride precipitated is dissolved in the solution obtained after treatment of the chloride liquor (or in any suitable solution), the copper being then obtained by cementation. C. A. KING.

Recovery of vanadium from solutions. R. H. STEVENS, G. C. NORRIS, and W. N. WATSON (E.P. 269,779 and 269,780, 23.8.26).—(A) Vanadium in solution is converted into the quinquevalent form, and precipitated, in the presence of a ferric salt, wholly or partly as ferric vanadate by the addition of a base, *e.g.*, lime. The precipitate is washed free from calcium sulphate by means of water or brine. (B) Preliminary purification of vanadium solutions is effected by adding a base to a solution containing quadrivalent vanadium. Silica, iron, alumina, and any other impurities which may be precipitated are removed, and the vanadium in solution is then converted into either the quinque- or ter-valent condition to facilitate its subsequent precipitation.

C. A. KING.

Conversion of lead sulphate into lead carbonate. S. C. SMITH (E.P. 272,053, 29.6.26. Cf. E.P. 239,257; B., 1925, 881).—In the manufacture of lead carbonate by passing carbon dioxide into a suspension of lead sulphate in ammonia solution, the product contains the minimum amount of lead sulphate if the carbon dioxide is introduced at such a rate that free carbon dioxide in the solution is avoided. W. G. CAREY.

Preparation of lithium carbonate. H. WEIDMANN, Assr. to ALLIED PROCESS CORP. (U.S.P. 1,623,513, 5.4.27. Appl., 12.5.26. Conv., 29.5.25).—Potassium chloride is added to a concentrated aqueous solution of lithium and potassium sulphates, and the precipitated potassium sulphate is separated from the solution. To the resulting solution rich in lithium chloride, potassium carbonate is added. Lithium carbonate in good yield is precipitated and potassium chloride regenerated, which can be used again in the first part of the process.

W. G. CAREY.

Manufacture of aluminium oxide. A./S. NORSK ALUMINIUM CO. (E.P. 252,399, 21.5.26. Conv., 23.5.25).—Silica-bearing slags containing calcium aluminate are pulverised and then leached and precipitated with a hot solution of sodium carbonate containing not more than 10 g./litre of free alkali hydroxide, to decrease the amount of silica dissolved and to produce a granular aluminium hydroxide precipitate. After filtration, the solution is treated with carbon dioxide to reconvert the greater part of the free caustic alkali into carbonate, so that the solution is available for further leaching.

W. G. CAREY.

Production of phosphorus. I. G. Farbenind. A.-G., Assees. of Chem. Fabr. Griesheim-Elektron (E.P. 247,219, 6.2.26. Conv., 6.2.25).—Hot crude gases from phosphorus furnaces are freed from dust by passing them through a filter chamber maintained at a temperature above the dew point of phosphorus by internal electrical heating or external heating by combustion gases, and, if desired, subsequently submitting them to an electrical dust-removing treatment. The filter chamber may be filled with the same materials as are used for charging the furnace, either in a moist or dry condition.

W. G. Carey.

Purification of hydrogen obtained by the interaction of phosphorus and steam. I. G. Farbenind. A.-G. (E.P. 262,455, 3.12.26. Conv., 4.12.25).—After condensing the bulk of the phosphorus acids the hydrogen, without previous cooling, is passed over active charcoal or silica gel, the substances adsorbed, *e.g.*, hydrogen phosphide, being oxidised by air during or after adsorption and the resulting oxides and acids of phosphorus being extracted with water or acids.

W. G. Carey.

Calcination of limestone and the like in vertical kilns fired with lean gas. Hauts Fourneaux & Acieries de Differdange-St. Ingbert-Rumelange, Soc. Anon., and E. Lavandier (E.P. 246,485, 21.1.26. Conv., 21.1.25).—The gas is burnt under pressure in mixing burners with sufficient air for complete combustion, the products of combustion being injected into the kiln through nozzles which localise a temperature zone in the kiln of at least 900°. W. G. Carey.

Manufacture of cyanides of alkali-forming metals. California Cyanide Co., Inc., Assees. of F. J. Metzger (E.P. 261,722, 14.10.26. Conv., 23.11.25).—See U.S.P. 1,573,732; B., 1926, 486.

Production of aluminium chloride. E. R. Wolcott, Assr. to Texas Co. (U.S.P. 1,633,835, 28.6.27. Appl., 2.2.21).—See E.P. 175,006; B., 1922, 252 a.

Filtration of solutions and the like. J. Blumenfeld (U.S.P. 1,633,621, 28.6.27. Appl., 28.7.24. Conv., 3.8.23).—See E.P. 228,814; B., 1925, 315.

Adsorbents (E.P. 271,564).—See I.

Ammonia from gases (U.S.P. 1,631,720).—See II.

Electrolytic apparatus (E.P. 271,685 and 271,726).—See XI.

Aurothiosulphate compounds (E.P. 261,048).—See XX.

VIII.—GLASS; CERAMICS.

Structure and constitution of glass. W. Rosenhain (J. Soc. Glass Tech., 1927, **11**, 77—97).—As a tentative working hypothesis, complex amorphous solids, such as glass, are considered as assemblages of atoms in which certain molecular groupings may occur with greater or less frequency, but in which the intra-molecular linkages need not be very different in kind or intensity from the other atomic linkages which exist, or come into being, when the substance is cooled down into the quasi-solid state. Formation of linkages will be limited by the inter-atomic distances and the angular relations between adjacent atoms. A portion only, probably small, of the inter-atomic linkages are operative, and the latent heat of fusion, representing the energy stored in unsaturated bonds, is largely retained in cooling and setting, so explaining the small heat evolution on cooling. With rise of temperature a point is reached above which each increment entails the breakage of certain linkages and the storage of energy or latent heat, and the heat added is used partly for this purpose and partly for increasing the amplitude of atomic thermal oscillation. The apparent specific heat of a vitreous substance should thus be greater than that of a crystalline substance of the same composition, and available data appear to bear out the point. There is an intermediate stage between elastic solid and viscous liquid in which rupture of bonds may occur for a time after application of stress, but with storage of sufficient energy to stop, after a time, further rupture; when stress is removed there will be gradual and incomplete return to the original configuration. The hypothesis fits the observed facts with regard to the slight thermal effect when glass is heated, and the variation of thermal expansion with temperature. The apparent modulus of elasticity should be decidedly higher than that of the same substance in crystalline form. Since solid diffusion is probably caused by "slip" mechanism, such cannot occur in cold amorphous solids, and diffusion at higher temperatures is probably due to viscous flow. The presence of molecular aggregates may result in neutral zones. A. Cousen.

Shrinkage of diaspore clays. I. S. M. Phelps (J. Amer. Ceram. Soc., 1926, 9, 659—666).—Three types of diaspore clays were studied in order to determine the nature and cause of the shrinkage which takes place in them after prolonged use. Under given conditions of time and temperature, the shrinkage varied inversely as the alumina content of the clays. The duration of the firing period had a marked effect upon shrinkage. The bonding (plastic) portion and the granular portion were separated by the aid of deflocculation and tested; considerable differences were observed in their porosity, shrinkage, and chemical composition. In the process of manufacture, diaspore refractories should receive sufficient heat treatment to produce constancy of volume within practicable limits. F. Salt.

Hypothesis explaining some characteristics of clay. J. Versluys (Proc. K. Akad. Wetensch. Amsterdam, 1927, **30**, 104—112).—A theoretical and mathematical paper in which it is shown by comparison with actual experimental curves that the tensile strengths of soils in their dependence on grain size and water content are directly calculable from the surface tension of the pendular water between the individual grains. The contracting effect of pendular water alone is insufficient to explain completely the peculiar phenomena exhibited by clay, *e.g.*, expansion and contraction on change in the proportion of water, for which purpose it is necessary to assume the existence of a power of expansion resulting from the possibly multimolecular film of adsorbed moisture which envelops each clay particle. Considering an ideal case in which two spherical particles are caused to approach with progressive displacement of the adsorbed film near the points of contact, it is shown that as the removal of adsorbed moisture proceeds the power

of expansion of the clay progressively increases. When clay dries out two forces are involved, the contracting force which increases as the water content decreases, and the expanding force, the equilibrium being continually shifted. An interpretation of the phenomena of quicksands is suggested, based on the probable behaviour of a mass of wet sand towards a sequence of physical disturbances. J. S. CARTER.

Clay analysis for control purposes. W. P. ECKDAHL (Chemist-Analyst, 1925, No. 45, 5—7; Chem. Zentr., 1927, I, 170).—A 0·5 g. sample is fused with sodium carbonate, dissolved in 60 c.c. of 1 : 1 hydrochloric acid and 12 c.c. of acetic acid, the undissolved silica filtered off, and iron oxide, alumina, and silica are precipitated by concentrated ammonia, after which the liquid is boiled and filtered. Lime and magnesia are determined in the filtrate. The precipitate is dissolved in hydrochloric acid, reprecipitated, filtered, ignited, and weighed, treated with hydrofluoric acid and weighed again. B. W. CLARKE.

Determination of ferrous iron in silicates. L. A. SARVER (J. Amer. Chem. Soc., 1927, **49**, 1472—1477).—The use of diphenylamine as an internal indicator in the titration of ferrous iron by potassium dichromate (cf. Knop, B., 1924, 404) is unaffected by the presence of hydrofluoric acid. The latter acid, however, decomposes potassium dichromate at higher temperatures so that the analysis of silicates by decomposition in presence of excess of dichromate, to eliminate oxidation by atmospheric oxygen, becomes impossible. A modified Pratt method of analysis is described (cf. Hillebrand, U.S. Geol. Survey Bull., 1919, No. 700, p. 203). 0·5 g. of silicate is boiled with 10 c.c. of either 12*N*-hydrochloric acid or 18*N*-sulphuric acid in a platinum crucible having a close-fitting transparent bakelite cover fitted with a bakelite funnel and an entrance tube. Air-free carbon dioxide is swept through the crucible for 10—15 min. during the boiling, and then 7 c.c. of 48% hydrofluoric acid are added through the funnel. After gently boiling for 10 min. the whole is cooled in a current of carbon dioxide, an excess of standard dichromate solution is added through the funnel, and the contents of the crucible are poured on to solid boric acid and titrated with ferrous iron solution using diphenylamine as indicator. The results are somewhat higher than those obtained in an ordinary crucible, but show good concordance. S. K. TWEEDY.

Effect of different forms of silica in porcelain bodies. H. HIRSCH (Ber. Deut. Keram. Ges., 1926, **7**, 49—88).—Tests were made on a porcelain body consisting of 50 pts. of kaolin, 20 pts. of Norwegian felspar, and 30 pts. of quartz. The raw quartz was added in a number of different forms: Scandinavian vein quartz, rock crystal, flint, geyserite, two kinds of quartz sand, and the washings from kaolin. These materials were examined mineralogically, and photomicrographs are given. The porcelain samples were tested for shrinkage, porosity, true and apparent sp. gr., cone m.p., coefficient of thermal expansion, tensile strength, resistance to the Brinell test, impact, electric breakdown, and translucency (photometric method); they were also examined in thin sections microscopically. Vein quartz produced by far the greatest translucency, especially in the hard porcelain series. Of the three strength tests, the Brinell test gave the most uniform and reliable results; the highest figures in these tests were obtained with prefired flint and quartz sand. These two materials, and the kaolin residues, also gave better results than quartz in the electrical test. Raw flint gave the lowest results in all these tests. It is concluded that crystalline quartz is only used on account of its effect upon translucency. Flint, quartz sand, and especially kaolin residues produce bodies which are stronger both mechanically and electrically. A study of thin sections revealed no direct relationship between microstructure and physical properties. The formation of mullite crystals was identified both microscopically and by chemical analysis. F. SALT.

Structure of the crystalline phases of porcelain. L. B. STROUTINSKY (J. Russ. Phys. Chem. Soc., 1927, **59**, 137—145).—Sillimanite subjected to *X*-ray analysis gives the following measurements: $a = 741$ Å.; $b = 7{\cdot}67$ Å.; $c = 5{\cdot}72$ Å. It is found to consist of 4 mols. of Al_2O_3,SiO_2. Andalusite also consists of 4 mols., and has the corresponding measurements: 7·76, 7·86, and 5·51 Å. Mullite gives figures pointing to $\frac{1}{2}Al_2O_3,SiO_2$, and, as this is impossible, the substance is either a mixture, or has been assigned an erroneous formula. E. ROTHSTEIN.

PATENTS.

Production of potash glass. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 245,785, 8.1.26. Conv., 8.1.25).—Potash glass is obtained by the use of potassium bicarbonate as a batch material; this substance is easily obtained pure, is non-hygroscopic, and promotes active mixing during melting. A. COUSEN.

Manufacture of silica glass articles. "QUARTZ & SILICE" (E.P. 266,711, 21.2.27. Conv., 24.2.26).—Addition of further material to vitreous silica articles is obtained by packing a mass of pulverulent silica in the desired region and fusing this to the original body by an apparatus containing, preferably, electrical resistance or high-frequency induction heating elements. Further parts may be so added to existing ware or articles of large dimensions built in successive stages. A. COUSEN.

Cement [ceramic] composition. C. E. KRAUS (U.S.P. 1,629,714, 24.5.27. Appl., 23.9.24. Cf. U.S.P. 1,509,406; B., 1925, 11).—Bentonite is added to kaolin or other clay to increase its plasticity when mixed with water. T. S. WHEELER.

Ceramic insulating material. H. H. SORTWELL, Assr. to STAR PORCELAIN CO. (U.S.P. 1,633,462, 21.6.27. Appl., 27.2.24).—A black porcelain insulating material consists of 45 pts. of black iron ore, 35 pts. of red-burning clay, 15 pts. of potter's flint, and 5 pts. of felspar. H. ROYAL-DAWSON.

Manufacture of an article of sillimanite-bonded granular material. M. C. BOOZE, Assr. to NORTON CO. (U.S.P. 1,616,525, 8.2.27. Appl., 20.5.21).—A ceramic article is made of graded super-refractory grains united by a small quantity of a synthetic sillimanite bonding material of the general formula $xAl_2O_3,ySiO_2$. Thus, crystalline alumina is bonded with a mixture of 7 pts. of

alumina (99% Al_2O_3) and 15 pts. of kaolin. The mixture after being moulded is fired at cone 16. H. Holmes.

Colourless Crookes glass. P. V. W. Gell, C. E. Gould, W. M. Hampton, and H. S. Martin, Assrs. to Chance Bros. & Co., Ltd. (U.S.P. 1,634,182, 28.6.27. Appl., 11.5.25).—See E.P. 256,737; B., 1926, 878.

Method and apparatus for producing a glass article [by severance]. E. C. R. Marks. From Libbey Glass Manuf. Co. (E.P. 272,308, 15.3.26).

Annealing ceramic wares (E.P. 248,394).—See I.

IX.—BUILDING MATERIALS.

Patents.

Conversion of slags into cement. S. Michelsen (E.P. 267,539, 10.3.27. Conv., 10.3.26).—Gypsum or other compounds containing sulphur are mixed with slag and heated so that the manganous oxide is partly or wholly converted into manganous sulphide. Limestone is then added if necessary in order to give the slag the required composition for use as cement. B. W. Clarke.

Calcining limestone (E.P. 246,485).—See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Transformation of retained austenite into martensite by stress. K. Honda and K. Iwasé (Sci. Rep. Tôhoku Imp. Univ., 1927, **16**, 1—8).—By measurements of changes in hardness and intensity of magnetisation, it has been shown that retained austenite in a quenched chromium steel is transformed into the stable martensite by cold hammering. On quenching plates of different thickness with oil and water, respectively, it has been found that, when the thickness of the plates is 3 mm. or more, water-quenched specimens are harder (*i.e.*, contain more martensite) than oil-quenched. This is in accordance with Mathews' observations (B., 1925, 807; 1926, 132) and also with Bain's view (Trans. Amer. Soc. Steel Treat., 1925, **8**, 14) that the internal stress produced by quenching promotes the transformation of retained austenite to martensite, the stress being greater in water-quenching. The contrary effect, however, is observed when the thickness of the plate is below 3 mm. In both methods of quenching the hardness increases with the thickness of the plate. When the steel is quenched in liquid air the hardness diminishes as the thickness of the plate increases. It is suggested that, although the supercooling of the quenched austenite facilitates transformation into martensite, the stress set up due to expansion may, after a certain point, prevent a further transformation of the remaining austenite, this stress naturally increasing with thickness. M. S. Burr.

Nitridation of steels. L. Guillet (Compt. rend., 1927, **184**, 1296—1299; cf. B., 1926, 410).—Tests on nitrided complex steels show increases in hardness particularly with chromium–tungsten–vanadium steels, pearlitic and martensitic nickel–vanadium steels, and nickel–manganese steels. The presence of vanadium, especially in nickel steels, has an important influence. Such steels are attacked by acid, alkali, and salt solutions to variable extents for different samples, and more so than ordinary cemented steel. The nitrogen is present probably as a complex nitride, and its concentration is highest on the surface of the metal. J. Grant.

Thermal changes in iron–manganese alloys, low in carbon. (Sir) R. Hadfield (Proc. Roy. Soc., 1927, **A**, **115**, 120—132).—The object of the investigation was the determination of the temperature of the critical magnetic changes in a series of iron–manganese alloys, practically free from carbon, containing 1·70—38·90% Mn. Magnetic transformations are confined to magnetic alloys with less than 16% Mn. The non-magnetic alloys above 16% show no magnetic transformations, no matter how heat-treated, or even when cooled to the lowest known temperatures. For all the magnetic alloys, a reversible magnetic transformation is found at practically the same temperature as the A2 magnetic change in iron (in this case, at 765—780° on heating, and at 767—776° on cooling). For alloys containing 6·75—14·30% Mn, a doubling of the magnetic transformation is observed on cooling, although not on heating. That is, the first recovery of magnetisation which takes place on cooling only holds over a limited range of temperature, the alloy becoming again practically non-magnetic, until finally, on further cooling, the full magnetic properties reappear and remain down to atmospheric temperature. The second critical point is progressively lowered with increase of manganese and decreases in intensity, and is not a fixed point for each alloy, but on repetition appears at a lower temperature. No such variation occurs in the position of the upper magnetic transformation. The magnetic strength of the alloys appears to become progressively weaker with increase of manganese. Heating and cooling curves of the inverse rate type are reproduced, and the relation between the magnetic changes and the thermal evolutions shown by the curves is discussed. It is suggested that the non-magnetic properties of the higher-percentage alloys may be due to chemical combination of the iron and manganese, the most probable formula for the resulting compound being Fe_5Mn. L. L. Bircumshaw.

Tensile tests on alloy crystals. I. Solid solution alloys of aluminium and zinc. II. Solid solution alloys of copper and zinc III. Conclusions. C. F. Elam (Proc. Roy. Soc., 1927, **A**, **115**, 133—147, 148—166, 167—169).—I. The results of tensile tests on single crystals of six alloys containing from 4·45 to 18·50% Zn showed that, with increasing percentage of zinc, the amount of extension before fracture tended to decrease while the breaking load increased. There was considerable variation especially in the elongation between crystals of the same composition. All the alloys show a well-marked yield point, particularly those with a high zinc content. Alloys containing 5—7·5% Zn usually show a double wedge-shaped fracture, as in the case of pure aluminium, whilst those containing over 10% Zn show a straight cleavage fracture (cf. A., 1925, ii, 954). Certain crystals containing 5—7·5% Zn showed an intermediate structure in which the double wedge was replaced by a single wedge asymmetric to the axis of the specimen. This may be due to the fact that, as the zinc content increases, the possible amount of extension

before fracture decreases, so that fracture may occur before the position for slip on two planes is reached. The results from both distortion and *X*-ray measurements with 10 and 15% zinc alloys indicate that the crystals deform by slip on an octahedral (111) plane in a direction represented by the normal to a (110) plane. This was confirmed by observation of slip-bands. There is also evidence that the 10% alloy crystal slipped on a second plane for about the last 10% of its extension. In many specimens the plane of fracture agreed very closely with the slip-plane, but in some the two made a small angle of from 10° to 20° with each other. Calculation of the shear stress on the slip-plane and the amount of shear showed that the resistance to shear increased considerably with increasing zinc content. The maximum increase in hardness occurs in the early stages of deformation, but the shear stress increases right up to the breaking-point.

II. Tensile tests have been carried out on a number of brass crystals containing about 70—80% Cu, which had been prepared by a method previously described (A., 1926, 1085). Distortion and *X*-ray measurements indicate that deformation occurs by slip first on the (111) plane in the (110) direction, and then on the (111) plane in the (011) direction. The most noticeable features of the distortion of these crystals are, firstly, the enormous extension (160%) before fracture, compared with copper, which rarely exceeds 60%, and, secondly, the uniformity of the crystal in its final condition, considering the abrupt changes in shape when the slip changes from one plane to the other. Calculations were made of the shear stresses on both planes; the shear stress at the beginning of the test is very little different from that of copper. The different octahedral planes in the crystal appear to be unequally resistant to shear, and it is suggested that slip tends to proceed more easily along a plane upon which it has once begun.

III. The results obtained by Rosbaud and Schmid (A., 1925, ii, 488) in their investigation of the effects of alloying on distortion in the case of zinc–cadmium and zinc–tin alloys are in general agreement with those described in the preceding investigation on the aluminium–zinc alloys. The fact that, in many respects, the results obtained with brass crystals appear to be directly opposite to those with aluminium–zinc crystals may possibly be ascribed to the different methods of preparing the crystals. It is suggested that the alloying atoms take up different positions when the crystals grow from the liquid from those occupied when the metal recrystallises in the solid, and it is probable that alloy crystals grown in the solid by a process of straining and heat-treatment are more uniform in structure than those grown from the liquid. L. L. Bircumshaw.

Cementation of aluminium and duralumin after double electrolytic deposition. J. Cournot and E. Perot (Compt. rend., 1927, **184**, 1250—1252; cf. B., 1927, 167).—The cementation of aluminium and duralumin coated electrolytically with copper and then with nickel, chromium, cobalt, or cadmium has been attempted by heating in a sand-bath or tubular electric furnace under varying conditions of deposition and cementation. Folds were produced at cementation temperatures above 575°, and sounder surfaces resulted from sand-bath treatment and double cementation. The total degree of penetration depends principally upon the copper deposit. The cemented zone consisted of a solid solution surrounded by a eutectic phase analogous with Al–Cu, and a few hard constituents produced by combination of the copper with the metal forming the outside deposit. J. Grant.

Influence of small quantities of bismuth, tin, lead, etc. on the structure and working properties of gold and gold alloys. L. Nowack (Z. Metallk., 1927, **19**, 238—244; cf. A., 1926, 896).—The most objectionable impurities in gold and gold alloys are bismuth, tellurium, and lead, less than 0·1% of which renders the metal unworkable owing to the segregation of bismuth, $AuTe_2$, and Au_2Pb along the grain boundaries. Prolonged annealing of metal containing these impurities serves to enhance their deleterious effect. More than 0·1% Sb renders gold brittle owing to the separation of the eutectic, but 0·1% Al, up to 1% Sn, and 2—3% Fe have little effect other than a slight hardening action. Characteristic photomicrographs of gold with various impurities are reproduced showing the modifying action of the impurity on the grain structure. A. R. Powell.

Recrystallisation and annealing of precious metal [silver–copper–gold] alloys. L. Sterner-Rainer (Z. Metallk., 1927, **19**, 149—153, 245—248).—The hardness of cold-rolled silver–copper alloys (80% Ag) increases slightly with rise of annealing temperature to 315°, then decreases rapidly. The tensile strength and elastic limit decrease slightly, and the elongation is increased by keeping the alloy in boiling water for 1—1·5 hr. The rate at which annealing takes place increases rapidly with rise of temperature above 315°, but even at 720° annealing is not completed in less than 80 min.; at 750°, however, the maximum ductility is obtained in 20 min., and further heating decreases it again. The best method of ascertaining when a sample of cold-rolled metal is completely annealed is to determine its elongation, as this property is more drastically affected by heat treatment than any other. Thus, after 60% reduction in thickness the metal has an elongation of 4·4%, which increases to a maximum of 36·2% at 720°. Similar behaviour is shown by 14-carat gold containing equal proportions of silver and copper. After a 10% rolling the tensile strength, elastic limit, and hardness increase on annealing at 350°, and, to a smaller extent, at 450°; with a 50% reduction by rolling, however, annealing above 300° causes a rapid softening. The hardening action of a low-temperature anneal is hardly noticeable with 18-carat gold, and does not occur with alloys of higher carat. A. R. Powell.

Determination of vanadium in iron minerals and rocks. V. A. Silbermitz and L. V. Rozhkova (J. Russ. Phys. Chem. Soc., 1927, **59**, 121—124).—A new colorimetric method for determining vanadium present in minerals and rocks which are soluble in acids or water is described. The pulverised rock is dissolved in nitric acid, the chlorides are precipitated with silver nitrate, and the organic matter is oxidised with potassium permanganate, excess of the latter being removed by the addition of hydrogen peroxide solution. Phosphoric acid is now added, the excess of hydrogen peroxide being destroyed by boiling, and then ammonium molybdate

solution. The colour obtained is compared with a standard chart prepared from standard solutions of ammonium vanadate. The method is quite as accurate as the more usual methods. E. Rothstein.

Synthetic testing for flotation. C. G. McLachlan (Trans. Canad. Inst. Min. Met., 1926, **29**, 251—280).—Laboratory experiments on the flotation of certain minerals by selective reagents, *e.g.*, oleic acid, aniline, showed that selective adsorption of oils is not the primary controlling factor in flotation. The forces involved are surface forces, and consequently the relative magnitude of these forces increases as the size of the mineral particle decreases. The electrostatic charge normally possessed by a mineral particle plays a very minor part. In certain cases of added electrolytes, it was evident that definite chemical interaction was necessary for successful working; *e.g.*, copper sulphate promotes the flotation of sphalerite by coating it with a film of copper sulphide. Similarly, the beneficial action of sulphuric acid in the treatment of certain zinc ores is due to copper associated with these ores going into solution as sulphate. The atomic arrangements of a number of sulphides, oxides, and carbonates were examined. In chalcopyrite and pyrite the faces of the unit crystal contain metal atoms only; in molybdenite, sulphur and metal atoms; whilst in sphalerite the zinc and sulphur atoms are interchangeable. In non-floatable minerals, *e.g.*, oxides of copper, zinc, arsenic, and members of the calcite group of carbonates, oxygen and not sulphur atoms are present in the crystal faces, and it is suggested that only surfaces which contain elements having an affinity for oxygen are capable of flotation. C. A. King.

Patents.

Heat-treatment of steel. N. D. Chopra and F. J. Bullen (E.P. 271,606, 1.4.26).—Steel is heat-treated in contact with furnace gases which have been treated with a mixture of an alkaline-earth oxide (10—50%) and carbonaceous material (4—20% of free carbon). An exhausted carburising mixture forms a suitable material. Steel turnings or other form of steel which exposes a large surface to the furnace gases may surround the article to be treated. C. A. King.

Treatment [to prevent corrosion] of iron and steel articles. Metals Protection Corp., Assees. of C. H. Humphries (E.P. 264,788, 23.8.26. Conv., 25.1.26).—Articles of iron or steel are subjected, with or without the aid of electrolysis, to a solution containing chromic acid so as to form a surface film containing iron and chromium oxides, and chromates. The coating formed is adherent and resistant to acid fumes, and forms a good surface for the after-application of paint or enamel. C. A. King.

Making grey-iron castings of any desired structure. Maschinenfabr. Esslingen (E.P. 260,990, 2.11.26. Conv., 6.11.25).—The total content of carbon and silicon in iron for a casting of definite thickness is selected from a graph showing the relation of structure with the wall thickness and the content of carbon and silicon. C. A. King.

Magnetisable material. E. Gumlich (E.P. 262,153, 30.11.26. Conv., 30.11.25).—An iron alloy of high magnetic permeability contains more than 40% Ni and 5—20% Mn. C. A. King.

Bearing metal. L. Sempell (E.P. 272,065, 4.8.26).—A bearing metal is made by fusing together 8—25% Sn, 1—4% of ammonium or calcium molybdate (1—2·5% of a 70% ferromolybdenum alloy), 0·5—2% Ni, 0·5—2% Cu, 13—20% Sb, the remainder being lead. C. A. King.

Bearing metal. P. Kemp and T. Kittl (U.S.P. 1,632,604, 14.6.27. Appl., 10.8.25. Conv., 23.8.24).—The alloy consists of 65—77% Pb, 3—14% Sn, 10—27% Sb, and 0·7—2·5% As. F. G. Crosse.

Treatment of zinciferous flue dust. F. Krupp Grusonwerk A.-G. (E.P. 265,558, 29.12.26. Conv., 4.2.26).—Zinciferous flue dust is heated in a rotary furnace at a temperature sufficiently high to sinter the material and at the same time volatilise the impurities, *e.g.*, arsenic, antimony, tin, cadmium, lead, and various acid radicals. The temperature in the furnace may approach the volatilisation point of zinc, and fluxes may be added to assist in the purification. C. A. King.

Acid extraction of metals such as zinc and vanadium. R. H. Stevens, G. C. Norris, and W. N. Watson (E.P. 269,777, 23.8.26).—An ore containing both a relatively easily and difficultly soluble metal is extracted with a dilute acid to dissolve the easily soluble metal. The residue, if necessary after being reground, is then leached with hot acid of suitable concentration, *e.g.*, for vanadium, preferably about 50 g. of sulphuric acid per litre, and the strongly acid solution is returned as leaching acid in the first leaching step. C. A. King.

[Desulphurising] treatment of nickel-containing mattes. H. Wade. From Internat. Nickel Co. (E.P. 271,282, 16.7.26).—Nickel or nickel–copper mattes are converted by blowing gases containing oxygen through the molten matte at 1380—1750°, and reducing the oxygen content of the blast during the later stage of blowing. Additional heat may be supplied by combustion above the bath in the converter, which is lined with fused alumina bonded with kaolin. C. A. King.

Copper–tin–nickel alloy. R. Ozlberger (E.P. 268,798, 31.3.27. Conv., 3.4.26).—A copper alloy containing about 7·3% Sn and 0·25% Ni. C. A. King.

Solders for aluminium and its alloys. E. C. R. Marks. From Vergo Aluminium u. Metallverwertungs-Ges.m.b.H. (E.P. 271,662, 29.6.26).—The solder consists of an alloy containing up to 70% Al together with tin, zinc, copper, iron, silicon, and lithium; *e.g.*, the constituents may consist of 450 pts. of aluminium, 370 pts. of tin, 180 pts. of zinc, 20 pts. of copper, 15 pts. of silicon, 10 pts. of lithium, and 5 pts. of iron. C. A. King.

Production of corrosion-resistant articles. Aluminum Co. of America, Assees. of E. H. Dix, jun. (E.P. 271,798, 28.3.27. Conv., 22.1.27).—An impure aluminium or aluminium alloy is protected from corrosion by casting it in contact with a plate or casing of corrosion-resistant aluminium or alloy. The latter may be in contact with a mould by which heat is removed at such a rate as to permit only surface fusion of the aluminium. The compound article may be pressed or otherwise hotworked to improve cohesion. C. A. King.

Manufacture of a light aluminium alloy. V. E. Hybinette (E.P. 271,597, 19.3.26. Cf. U.S.P. 1,579,481; B., 1926, 496).—A primary alloy containing 60—75% Al together with nickel and metals of the chromium group is prepared and introduced into molten aluminium to give a final alloy containing not less than 94% Al, 0·5—3% Ni, and 0·1—0·75% of metals of the chromium group. As hardening elements may be introduced 0·1—0·75% Cu (Mn) and 0·1—1% Fe. C. A. King.

Bright-annealing [muffle] furnace. Siemens-Schuckertwerke G.m.b.H. (E.P. 262,433, 1.12.26. Conv., 3.12.25).—The time taken by the material annealed to cool down is considerably reduced by arranging axially in the muffle a tube, closed at both ends in a heat-insulating manner, which can be opened at both ends while the material is cooling so that air from outside the muffle enters the tube. If desired the tube may be coated internally with enamel. J. S. G. Thomas.

Process and apparatus for splitting up metal alloys into their various component kinds of crystals by centrifuging. K. Schmidt (E.P. 271,293, 10.8.26).—Metal alloys, particularly easily fusible alloys, are introduced into a centrifuge of the basket type, the temperature being so adjusted that one type of crystal present in the alloy is in the solid state while all the remaining components are liquefied. This condition is maintained until the solid and liquid constituents have been separated by centrifuging. C. A. King.

Furnace for smelting and refining iron, steel, and other metals. P. Kühn (E.P. 248,012, 23.2.26. Conv., 23.2.25).—In a hearth furnace having regenerators for preheating air for combustion directly connected to each end of the hearth, a rich fuel is injected through openings in the crown directly on to the charge and into the current of air entering from the regenerator. C. A. King.

Treatment of zinc-bearing ores and metallurgical products. H. W. Gepp, H. Hey, G. Rigg, R. H. Stevens, and R. T. D. Williams, Assrs. to Electrolytic Zinc Co. of Australasia, Ltd. (U.S.P. 1,634,100, 28.6.27. Appl., 30.6.23. Conv., 1.8.22)—See E.P. 201,918; B., 1924, 679.

Heat-resisting alloy. G. R. Brophy, Assr. to General Electric Co. (U.S.P. 1,633,805, 28.6.27. Appl., 17.6.21).—See E.P. 179,306; B., 1922, 505 a.

Alloy. W. E. Ruder, Assr. to General Electric Co. (U.S.P. 1,633,826, 28.6.27. Appl., 7.6.23. Renewed 29.4.27).—See E.P. 216,389; B., 1924, 637.

Annealing, carburising, bright-annealing, and other heat-treatment furnaces. T. R. Speck (E.P. 271,918, 22.1. and 14.10.26)

[Sealing means for] electric smelting furnaces. A.-G. Brown, Boveri & Cie. (E.P. 263,872, 31.12.26. Conv., 2.1.26).

Annealing metals (E.P. 248,394).—See I.

Rustproofing oils (U.S.P. 1,630,101).—See II.

XI.—ELECTROTECHNICS.

Patents.

Electrolytic apparatus and electrodes therefor. W. G. Allan (E.P. 271,685, 4.8.26).—In an electrolytic cell for the decomposition of water, electrodes and their separating diaphragms are made of a sinuous or zig-zag shape, the electrodes being secured mechanically and electrically to opposite cell walls, whilst the diaphragm is supported by a holder carried by the cell ends, so that the total electrode area is large relative to the projected area and the current paths are of large total cross-sectional area. J. S. G. Thomas.

Electrolytic apparatus and electrodes therefor. J. P. Scott (E.P. 271,726, 27.10.26).—In an electrolytic cell for the decomposition of water, using relatively high current densities, sinuous electrodes are arranged longitudinally within the cell, and are supported in co-operating pairs, whilst a pervious diaphragm is arranged between the members of each pair, so that evolved gases are formed in separate compartments and pass out at the top of the cell. J. S. G. Thomas.

Gaseous electric conduction apparatus [rectifier]. Raytheon Manuf. Co., Assees. of C. G. Smith (E.P. 263,108, 15.11.26. Conv., 21.12.25).—An electric discharge device operating by conduction between electrodes within an enclosure contains two gases, one of which, *e.g.*, cæsium or other alkali metal or alkaline-earth metal, ionises more readily than the other, *e.g.*, helium, which is preferably inert with respect to the alkali vapour. The more easily ionisable gas is ionised by light radiation from a heated filament for starting purposes. J. S. G. Thomas.

Incandescence lamp. O. V. Maurer (U.S.P. 1,630,128, 24.5.27. Appl., 17.7.25).—An incandescence lamp containing a tantalum filament is filled with pure argon at atmospheric pressure. T. S. Wheeler.

Electric furnace for heating gases at high temperatures. A.-G. Brown, Boveri & Cie. (E.P. 264,849, 20.1.27. Conv., 22.1.26).—Long, narrow, refractory insulating tubes through which the gas to be heated passes, and which contain nickel-chromium heating spirals, are embedded in passages in a refractory stone filling of a steel tube provided with end covers serving as supports, and which carry connexions for the gas inlet and outlet pipes. J. S. G. Thomas.

Electric arc welding. British Thomson-Houston Co., Ltd. From General Electric Co. (E.P. 272,067, 7.8.26).—Methyl alcohol or acetone etc. is dissociated in the arc so as to produce an atmosphere composed substantially of carbon monoxide and hydrogen surrounding the arc and the molten portion of the work. J. S. G. Thomas.

Electrical resistance thermometer. A. Bursill, and Electroflo Meters Co., Ltd. (E.P. 271,921, 27.1.26).—A resistance element is supplied with alternating current, and is so connected that variations of current due to variations of its resistance cause variations of current in a moving coil disposed in the field of an alternating current electromagnet energised from the same source as the resistance element. J. S. G. Thomas.

Electrical insulating compound. A. E. L. Scanes, and Metropolitan-Vickers Electrical Co., Ltd. (E.P. 271,979, 22.3.26).—A plastic or fluid electrically insulating material, *e.g.*, material of the nature of pitch,

is intimately mixed with a substance or mixture of substances, *e.g.*, ammonium sulphate, ammonium phosphate, and carbon tetrachloride, which when heated evolve a gas (or gases) which precludes combustion. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 186,861, 3344 of 1906; also in pursuance of Sect. 8, Sub-sect. 2, to E.P. 252,856.] J. S. G. THOMAS.

Treatment of natural fibrous material for use in or as electric insulating material. STANDARD TELEPHONES AND CABLES, LTD. From WESTERN ELECTRIC CO., INC. (E.P. 271,948, 4.3.26).—Fibrous material is freed from extraneous ionisable matter by washing for 1—3 hrs. with water substantially free from such matter at 80—100° and subsequently treating the washed material with a solution of an electrolyte, *e.g.*, calcium hydroxide, which is irreversibly adsorbed by the surfaces of the fibres. J. S. G. THOMAS.

Incandescence lamp. G. R. FONDA, Assr. to GENERAL ELECTRIC CO. (U.S.P. 1,632,647, 14.6.27. Appl., 24.1.25).—See E.P. 246,516; B., 1926, 413.

[Electrode economiser for] electric furnaces. SIR W. G. ARMSTRONG, WHITWORTH & CO., LTD., and E. WINDLE (E.P. 271,739, 12.11.26).

[Contacts for self-baking] electrodes for electric furnaces. NORSKE AKTIESELSKAB FOR ELEKTROKEM. IND. (E.P. 262,722, 3.8.26. Conv., 9.12.25).

Electrolytic [pressure] system [for the generation of gases]. J. E. NOEGGERATH (E.P. 245,119, 18.12.25. Conv., 19.12.24).

[Electrodes for] gas-filled discharge tube. H. WADE. From N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 269,657, 20.1.26).

Selenium and like cells. FOXGROVE MACHINERY CO., LTD., and F. GROVER (E.P. 272,126, 31.1.27).

Treatment of sludge acid (U.S.P. 1,630,074).—See II.

Magnetisable material (E.P. 262,153).—See X.

Sterilising water (E.P. 271,721).—See XXIII.

XII.—FATS; OILS; WAXES.

Separation of liquid and solid fatty acids by Twitchell's method. A. STEGER and H. W. SCHEFFERS (Rec. trav. chim., 1927, **46**, 402—408).—The various methods of separating liquid and solid fatty acids by means of the differences in solubility of their metallic salts in organic solvents are criticised owing to the disturbing influence of their mutual solubilities, and, in the case of the salts of bivalent metals, to the formation of mixed saturated–unsaturated salts. The method recommended is Twitchell's modification (A., 1921, ii, 662) of Varrentrapp's lead salt–ether method, using alcohol as the solvent. It is concluded from a large mass of experimental data that the separation is strongly influenced by the nature of the solid fatty acids, stearic acid separating better than palmitic acid, and the latter better than a mixture of equal parts of the two. The nature of the unsaturated acids appears to have much less effect. Unsaturated solid acids such as elaidic behave mainly as saturated acids with a low m.p., but the separation is less complete. E. HOLMES.

Addition of iodine to unsaturated oils, fats, and fatty acids in an organic solvent. II. Determination of a mixture of oleic and elaidic acids. III. J. P. K. VAN DER STEUR (Rec. trav. chim., 1927, **46**, 409—413, 414—416).—II. In extension of earlier work (B., 1927, 494) a method is developed whereby it is possible to determine the amounts of oleic and elaidic acids in a mixture of the two, higher unsaturated acids being absent. A method of calculating equilibrium constants for various mixtures is given, starting from the values 94·7 and 5·0, at 0°, for oleic and elaidic acids, respectively. These exhibit good agreement with the observed values, and are best shown graphically for the purposes of the determination. Further values are given for the equilibrium constants at 19·5°, oleic acid 26·3, elaidic 2·0, linoleic 17·1, erucic 28·8, and brassidic 1·8, the first two showing the enormous influence of temperature on this constant. III. Equilibrium between solutions of iodine and unsaturated fatty materials in benzene is dependent on temperature. The equilibrium constants of the various compounds examined in benzene solution bear the same relation to one another, but are approximately one third of the values obtained in carbon tetrachloride solution, both at 19·5°. Comparison of the values for peanut oil and its fatty acids shows that the glycerol linkings have no influence on the equilibrium point. E. HOLMES.

Grape seed oil. J. F. CARRIÈRE and R. CAMPREDON (Chim. et Ind., 1927, **17**, 723—728).—The quality and yield of oil depend on mode of collection, degree of maturity of grape, and on climate and soil where cultivated, being 9—12% for fresh pips or 15—17% in the dry state. As 100 kg. of residue (45% of water) arising from the grapes give 20% of pips (40% of water) and 1 hectolitre of wine corresponds to only 12 kg. of residue, it follows that scarcely 200 g. of oil per hectolitre of wine are produced. It is economical to extract the oil on the spot either by crushing or solvent extraction. The residue, after fermentation of the grape juice, is treated in continuous presses to separate the seeds or pips from the pulp, the seeds being then dried until a humidity of only 20—25% is attained. Works operating day and night treat 5600—6000 kg. of seeds per 24 hrs., *i.e.*, extract 7—8 hectolitres of oil. When freshly prepared from seed of good quality the proportion of free acidity in the oil is low (0·44—4·7% for one factory). The residue of the industry, *i.e.*, the spent seed, is an excellent fertiliser containing 2% N, 0·5% P_2O_5, and 0·7% K_2O. H. M. LANGTON.

Prevention of autoxidation of olein. A. BAG and W. NOWIKOV (Chem. Umschau, 1927, **34**, 175—176).—The tendency for olein and various unsaturated fatty oils to undergo autoxidation has been examined by determining, in the Mackey apparatus, the rise in temperature of wadding impregnated with the test liquid. The effect of the addition of small amounts of amines, aldehydes, organic acids, phenols, and alcohols has been determined, and it is shown that, whereas olein and cottonseed oil attain a temperature of 140° in from

1 to 2 hrs., on the addition of 1% of β-naphthol they reach only 100° after 4 hrs. These results have been substantiated on the technical scale. It is also shown that the addition of 1% of β-naphthol to linseed oil prevents polymerisation and oxidation for at least two years, the viscosity and iodine value of the sample being unaltered. The action is regarded as negative catalysis. E. Holmes.

Cresol–soap systems. II. Gelatinisation tendency of cresol–soap systems. S. Jenčič (Kolloid-Z., 1927, **42**, 168—174. Cf. B., 1927, 494).—Sodium salts of the fatty acids show a pronounced lyophilic character towards cresol in absence of water. At the ordinary temperature the gelatinisation tendency of cresol–soap systems depends on the nature of the fatty acid, rising with increase of mol. wt. (for acids of even number of carbon atoms). The sodium salts of the lower members of the acetic acid series, although crystalloidal in water, give gels in cresol. The gelatinisation tendency of sodium salts of unsaturated fatty acids with 18 or more carbon atoms and 1 or 2 double linkings is lower than that of saturated fatty acids containing the same number of carbon atoms. Sodium ricinoleate possesses the lowest tendency to gelatinise. E. S. Hedges.

Patents.

Bleaching wool fat. I. Lifschütz (G.P. 438,292, 14.10.22. Addn. to G.P. 324,667; B., 1920, 790 a).—Products obtained by the partial or complete saponification of wool fat by the process described in the prior patent are bleached by exposure in a finely-divided form to the action of atmospheric oxygen. Bleaching may be accelerated by the addition of other oxidising agents or of oxygen carriers. L. A. Coles.

Deodorising, clarifying, and dehydrating vegetable and animal oils and fats. E. Foray (Addn. No. 31,090, 2.10.25, to F.P. 601,919; B., 1926, 987).—Sulphuric acid is added to a mixture of the oil or fat with water, zinc dust, and wood charcoal or decolorising charcoal, and when reaction has ceased the aqueous liquor is removed and the fat is washed with water until neutral. L. A. Coles.

Removal of suspended matters from oils and organic solvents. H. Hey (U.S.P. 1,633,941—2, 28.6.27. Appl., [a], 26.11.21, [b], 11.3.24. Conv., 16.12.20).—See E.P. 176,540; B., 1922, 334 a.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Testing the mechanical properties of paint and varnish films. I. H. Wolff and G. Zeidler (Farben-Ztg., 1927, **32**, 2135—2136).—An apparatus is described by the use of which the elasticity and tensile strength o. detached paint and varnish films may be determined under constantly increasing load, attained by flow of water from an upper to a lower chamber, the film being suspended between them. The apparatus records and shuts off automatically. S. S. Woolf.

Accelerated paint testing. M. Schulz (Farben-Ztg., 1927, **32**, 2128—2133).—The functions of the components of accelerated weathering cycles are discussed, and descriptions and photographs of actual cycles in use in various German laboratories are given. In tests in which the concordance between the results of accelerated and ordinary exposure tests is well illustrated, it is established that, in the protection of iron against rust, red lead–oil primers are superior to nitrocellulose primers, whilst sand-blasting the iron surface decreases the protective action of paint applied thereto. S. S. Woolf.

Influence of plasticising on the mechanical-elastic properties of artificial and natural plastic substances. II. Artificial resins. O. Manfred and J. Obrist (Kolloid-Z., 1927, **42**, 174—180).—The "streaming effect" described in a previous paper (A., 1927, 514) has been further examined. The character of artificial resins is less dependent on their chemical constitution than on their state of dispersity and re-aggregation. Stirring during the process of aggregation has a favourable influence on the orientation of the rod-like particles. The modulus of elasticity of a number of artificial resins has been determined. E. S. Hedges.

Grape seed oil. Carrière and Campredon.—See XII.

Patents.

Water paint or distemper. C. J. Millar (E.P. 271,149, 18.2.26).—On applying one coat of a distemper consisting of a loosely-bound emulsion of water, varnish or oil, an organic agglutinant, and pigments, to absorbent or non-absorbent surfaces, partial separation ensues, an even coating being furnished by one portion, whilst the remainder segregates to form irregular designs of decorative value, the appearance of wall paper being obtained. A preferred composition is:—140 pts. of a casein solution, 140 pts. of varnish, 260 pts. of pigment, and 100 pts. of water. S. S. Woolf.

Production of low-viscosity lacquer and film. E. M. Flaherty, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,629,999, 24.5.27. Appl., 23.5.21).—The viscosity of a solution of pyroxylin in the usual solvents is greatly reduced by allowing it to remain for several weeks with a small quantity of sodium acetate dissolved in methyl alcohol. The resulting solution, though containing 25—30% of pyroxylin, can be used without thinning in the preparation of lacquers, which give films of considerable thickness. T. S. Wheeler.

Manufacture of water-soluble condensation products. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 248,782, 8.3.26. Conv., 9.3.25).—A halogenated simple or mixed aliphatic ketone or ether or a halogenated aliphatic aldehyde is condensed with a phenol in the presence or absence of a condensing agent, and the product, after washing, is rendered water-soluble by treatment with concentrated sulphuric acid. S. S. Woolf.

Manufacture of condensation products from carbamide or its derivatives and formaldehyde. Soc. Chem. Ind. in Basle (Ges. für Chem. Ind. in Basel) (E.P. 249,101, 1.3.26. Conv., 10.3.25).—Sufficient carbamide is added to the initial water-soluble condensation product of 1 mol. of carbamide with at least 2 mols. of formaldehyde, at any stage of the concentration, to bring the proportion to not less than 1·05 mols. of carbamide to 2 mols. of formaldehyde. The mixture is

converted into the solid hard final condensation product in the presence of an acid catalyst, which may be added at the same time as the additional carbamide or at a different stage of the concentration. S. S. WOOLF.

Manufacture of transparent and colourless condensation products of carbamide and solid polymerides of formaldehyde. F. E. K. STEPPES and H. O. TRAUN (H. TRAUN & SÖHNE) (E.P. 271,264, 22.6.26).—Solutions containing up to 75% of solid matter are obtained by dissolving carbamide in aqueous solutions of formaldehyde of strength exceeding 40% (prepared by the use of reagents which promote dissolution in water of solid polymerides of formaldehyde, *e.g.*, ammonia, alkalis, formates, acetates, sulphites, hydrochloric acid, aromatic sulphonic acids). On boiling these carbamide-paraldehyde solutions for a short while in an open vessel, viscous condensation products are obtained that may readily be hardened by further heating at 100°, the difficulties customarily experienced in the removal of water and the tendency to crack on hardening being obviated. S. S. WOOLF.

Production of a polymerised vinyl chloride modification [β-caouprene chloride]. L. A. VAN DYK (E.P. 255,837, 29.6.26).—If the polymerisation of vinyl chloride by means of ultra-violet rays or sunlight, with or without a catalyst (*e.g.*, a soluble lead salt), be continued beyond the acetone-soluble or α-stage, a new (β-) modification insoluble in acetone but soluble in monochlorobenzene is obtained, before conversion into the insoluble (γ and δ) forms. The β-polymerised vinyl chloride (β-caouprene chloride) may also be obtained from the γ and δ forms by heating them with aniline, quinoline, etc. Monochlorobenzene solutions of β-caouprene chloride to which is added a plasticiser, *e.g.*, a dichlorobenzene etc., yield flexible, transparent, and non-inflammable films on drying. S. S. WOOLF.

Production of a polymerised vinyl chloride [β-caouprene chloride]. L. A. VAN DYK (E.P. 260,550, 26.7.26).—The β-caouprene chloride described in E.P. 255,837 (cf. preceding abstract) is obtainable from the α-compound by prolonged action of ultra-violet rays and/or heat. Vinyl chloride is polymerised in a closed quartz vessel by means of sunlight or ultra-violet rays to α-caouprene chloride, and the unchanged vinyl chloride is distilled off. The α-compound is isolated by dissolution in acetone, and is submitted to further action of the rays or is heated at 50—135° until soluble in chlorobenzene but no longer soluble in acetone.
C. HOLLINS.

Filter stones (E.P. 251,964—5).—See I.
Lead carbonate (E.P. 272,053).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Spiral structure of rubber. H. FEUCHTER (Kautschuk, 1927, 98—101, 122—124).—The structure of rubber is regarded as involving the polymerisation of isoprene or dimethyloctadiene into concentrically superposed spherical polyprene shells; the latex globule constitutes a single shell aggregate or co-molecule (cf. B., 1927, 148). It is suggested that each shell possesses residual affinity localised at a point, and that the attachment of shell to shell occurs by means of these residual affinities, a succession of such bonds over a number of shells following a continuous spiral course. In ordinary rubber the globules are attached to one another by localised but movable surface valencies or co-valencies. The fibrous character of stretched rubber and the possibility of retraction after stretching are explained with the assumption of a reversible anisotropic phase-crystallisation. D. F. TWISS.

Fibre structure and Röntgen interference of stretched synthetic rubber. L. HOCK (Kautschuk, 1927, 125—126).—Contrary to a recently expressed view that so-called "synthetic rubber" is not really rubber because it fails to show the *X*-ray interferences exhibited by natural rubber, the former under suitable conditions actually does reveal the fibrous character and the interference phenomena characteristic of rubber.
D. F. TWISS.

Sulphur and theory of accelerators of vulcanisation [of rubber]. P. SCHOLZ (Kautschuk, 1927, 101—103, 127—128).—Addition of small proportions of various organic accelerators of vulcanisation to molten sulphur expedites the attainment of equilibrium between the allotropic forms of the element. It is assumed therefore that such substances accelerate vulcanisation either by facilitating the conversion of Sλ into more chemically active Sπ or Sμ, or that this change in the sulphur induces a parallel transformation of the rubber into a more active form (cf. J.S.C.I., 1921, 48 T). D. F. TWISS.

Ultramicroscopical studies on the theory of vulcanisation. H. DANNENBERG (Kautschuk, 1927, 104—105, 128—130).—The microscope and ultra-microscope are applied to the examination of the behaviour of mixtures of rubber (or of paraffin oil) with sulphur, zinc oxide, and an organic accelerator, when heated and subsequently cooled. When heated with rubber, well dispersed sulphur dissolves without melting. Further heating at 120° causes separation of a colloidal substance, probably Sμ; this change appears to be coincident with incipient vulcanisation, but the deposition does not continue with longer heating. It is believed that vulcanisation is dependent on the change Sλ→Sμ, and that vulcanisation accelerators are substances capable of expediting this change. Although the mechanism of vulcanisation may be explained by Sμ being much more chemically active towards rubber, or by the formation of colloidal sulphur inducing "polymerisation" of the rubber, the view favoured is that vulcanised rubber is a dispersion of Sμ in raw rubber. D. F. TWISS.

Production of rubber articles from various preserved [rubber] latices and prevention of the development of tackiness. R. DITMAR (Gummi-Ztg., 1927, 41, 1688—1689).—For the manufacture of cold-vulcanised articles from preserved latex, ammonia is a better preservative than formaldehyde; caustic soda is very unsatisfactory. The undesirable development of tackiness in cold-vulcanised articles on storage can be prevented by soaking in a 35% solution of formaldehyde after vulcanisation, by giving this treatment before vulcanisation and either before or after wiping with benzene, or by freely applying colloidal clay before or after vulcanisation. D. F. TWISS.

Patents.

Vulcanised products for use in the manufacture of rubber goods, of paper or paper boards, or for coating fabrics. F. Kaye (E.P. 271,553, 23.2.26).—Emulsified oils or fats are treated with a vulcanising agent, *e.g.*, a sulphide or a polysulphide, or are mixed with rubber latex and then vulcanised. Substances may also be added to accelerate vulcanisation. The products may be allowed to gel, with the aid of a coagulant, *e.g.*, aluminium sulphate, if desired, and then sheeted, with or without the incorporation of fillers. Alternatively, the products may be beaten into paper pulp; a coagulant is then added and the mixture formed into paper or paper board. D. F. Twiss.

Manufacture of moulded rubber articles from latex. J. McGavack, Assr. to Naugatuck Chemical Co. (U.S.P. 1,629,924, 24.5.27. Appl., 24.3.26).—Rubber latex, with or without vulcanising agents and fillers, is mixed with an aqueous emulsion of pine tar and allowed to set in a mould. The product is dried first at ordinary, and then at higher, temperatures.

T. S. Wheeler.

Thickening and stabilising latex. M. C. Teague, Assr. to American Rubber Co. (U.S.P. 1,634,124, 28.6.27. Appl., 30.9.25).—See E.P. 235,232; B., 1925, 729.

XV.—LEATHER; GLUE.

Rendering gelatin insoluble. G. E. Holden (J. Soc. Dyers and Col., 1927, 43, 194—196).—About 17·5% of soluble gelatin is converted into an insoluble form by heating at 120° for 8 hrs., the same proportion of insoluble gelatin being obtained when formaldehyde is used as fixing agent. The insoluble gelatin possesses approximately the same empirical composition as the soluble and the original gelatin. Both the insoluble and the untreated gelatin possess approximately the same absorptive powers for tannic acid, but the insoluble form showed increased affinity for basic dyestuffs when applied to cotton in association with tannic acid. It is probable that the altered dyeing properties are due to the conversion of a part of the original gelatin into an insoluble anhydride. The insoluble formo-gelatin compound formed with formaldehyde is decomposed by repeated washings with boiling water, and leaves fixed in an insoluble state approximately the same percentage of gelatin as is produced by the agency of heat. Samples of gelatin used in this work had the composition: C 50·21%, H 6·35%, N 17·72%, ash 1·85% (French); and C 50·42%, H 6·75%, N 17·61%, ash 1·95% (English). R. Brightman.

Patents.

Manufacture of tanning agents. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 260,543, 16.4.26. Conv., 28.10.25).—A salt of aluminium is mixed with a salt (other than a nitrite) of an alkaline-earth metal or of another bivalent metal, *e.g.*, zinc or magnesium. Thus, aqueous solutions of aluminium sulphate and magnesium sulphate are mixed, then concentrated to a paste, and the product is dissolved as required for tanning purposes. It produces a white, soft leather. D. Woodroffe.

Manufacture of coloured dressings for leather. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 248,767, 5.3.26. Conv., 6.3.25.)—A pigment dye (*e.g.*, Hansa Yellow) is intimately ground with a substance of high viscosity capable of gelatinising cellulose esters (*e.g.*, tricresyl phosphate) and combined with a substance not capable of gelatinising cellulose esters (*e.g.*, butyl acetate), the mass being mixed with a celluloid or nitrocellulose varnish.

D. Woodroffe.

Manufacture of artificial horn. British Glues and Chemicals, Ltd., R. Duncalfe, and H. J. Cotes (E.P. 271,221, 13.4.26).—A nitrogenous substance (*e.g.*, chrome leather from which the glue-forming matter has been wholly or partially removed) is screened, pressed to reduce the water content, mixed with an absorbent binding material (*e.g.*, saponified resin, casein, calcium or magnesium chlorides) to which an organic acid may be added, and moulded. The product is then treated with a formaldehyde solution, washed, and finally dried. D. Woofroffe.

[Pressing devices for use in the] production of artificial horn and goods made therefrom. O. Manfred (E.P. 269,761, 3.7.26).

XVI.—AGRICULTURE.

Effect of large applications of potassium and chlorine on the growth, leaf colour, and yield of potatoes. Chlorosis as a disturbance of ionic equilibrium in plants. K. Maiwald (Z. Pflanz. Düng., 1927, A9, 57—98).—The pure salts, potassium chloride and sulphate, sodium sulphate, and calcium chloride, given separately in rather excessive amounts, with and without nitrogen as ammonium nitrate, produced characteristic effects on the growth of potatoes in vegetation experiments. It was clear that with potassium and sodium sulphates the effects were due solely or predominantly to the cations, with calcium chloride solely to the anions, and that with potassium chloride both anions and cations influenced the condition of the plants. Special attention was paid to leaf colour, and the plants were graded according to the depth of colour of the leaves. The classification adopted was shown to accord well with the actual content of chlorophyll, as determined by the method of Willstätter and Stoll. The influence of excess of chlorine ions caused a reduction in chlorophyll content sufficiently great to be classed as incipient chlorosis. Excess of potassium or sodium ions alone effected a reduction in leaf colour, as compared with normal plants, of about 25%, chlorine ions alone about 70%, and potassium and chlorine ions together, about 60%. When, however, ammonium nitrate was given in addition, this reduction in colour was converted into an increase over the normal of 25—35%. The author discusses his results in some detail in relation to the literature on the absorption of ions by plants and their effects, and considers that not only chlorosis, but a whole series of phenomena concerned with plant metabolism, can be attributed to the alteration of the equilibrium between physiologically important ions in the plant. The behaviour of the plants in regard to selective absorption of ions was closely similar to that

observed by Hoagland in experiments with barley in water culture (cf. B., 1924, 143). C. T. GIMINGHAM.

Conception of the nutrient requirement of a soil and its determination. R. MEYER (Z. Pflanz. Düng., 1927, **A9**, 99—109).—The necessity for a clearer conception of the meaning to be attached to "the nutrient requirement of a soil" is pointed out. The idea must be considered from an economic point of view, and involves the factor of yield. It follows that Mitscherlich's method is the only one available for an exact determination, since it alone takes yield into account. It is particularly recommended that the yield factor be investigated simultaneously in the same experiment for more than one nutrient. C. T. GIMINGHAM.

Comparative manuring trials on arable land and pastures. M. POPP, W. FELLING, and R. FLOESS (Fests. Versuchs-Stat. Oldenburg, 1926, 66; Bied. Zentr., 1927, **56**, 205—207).—The potash content of hay in pastures is only of value as a measure of the available supplies of potash in the soil when nitrate and phosphate are present in optimum relative proportions. In cropping trials, basic slag gave better results than Algerian phosphate. The difference between the crop increases following the use of these two fertilisers was less with oats than with potatoes. Larger dressings of Algerian phosphate gave greater proportional crop increase with potatoes than with oats. Using similar amounts of phosphate as basic slag, the reverse proved to be the case. A. G. POLLARD.

Action of ammonium sulphate and of sodium nitrate on acid sandy soils. D. MEYER (Mitt. Deut. Landw.-Ges., 1926, 740—742; Bied. Zentr., 1927, **56**, 208—209).—Increased soil acidity following the use of ammonium sulphate is not satisfactorily remedied by liming. The use of the nitrates of sodium and calcium as fertilisers on light sandy soils is to be recommended. A. G. POLLARD.

Manuring with phosphates. O. WOLTE and R. LEONHARDS (Mitt. Deut. Landw.-Ges., 1926, 701—706; Bied. Zentr., 1927, **56**, 207—208).—Extensive experiments demonstrate the increased need of German soils for phosphate, as an outcome of the shortage of supplies of high-grade basic slag. Deficiencies of phosphate in soils are emphasised during dry periods. In most fertiliser trials adequate supplies of phosphate must be maintained in the soil if reliable results are to be obtained. A. G. POLLARD.

Action of various phosphatic manures. M. POPP and J. CONTZEN (Fests. Versuchs-Stat. Oldenburg, 1926, 48; Bied. Zentr., 1927, **56**, 251—254).—Tests of the value of phosphates with and without lime on acid soils are recorded. On sandy soils lime increased the intake of phosphate by pasture grasses. A similar effect on peas was not observed. Crop increases following the use of basic slag on sandy soils were less when lime was used in addition. With Rhenania phosphate the reverse was the case. Colloidal phosphates were more effective on acid soils. Lime decreased the effectiveness of tri- and di-calcium phosphates, the effect being more marked in the case of the former. Lowered crop increases resulted from the use of lime with magnesium ammonium phosphate, the effect being due to changes in soil reaction rather than to an adverse lime–magnesia ratio. The activity of superphosphate on sandy soils was increased by the use of lime. On moorland soils results were irregular, and frequently the reverse of those obtained on sandy soils. The intake of phosphate by plants appeared to be facilitated on moorland soils. A. G. POLLARD.

Accuracy of the various methods of measuring concentration of hydrogen ions in soil. C. OLSEN and K. LINDERSTRØM-LANG (Compt. rend. Trav. Lab. Carlsberg, 1927, **17**, 1—27).—Comparison of the hydrogen electrode, quinhydrone electrode, and colorimetric methods for determination of the p_H of soils, based on the study of about 100 soil samples, leads to the conclusion that measurements made on soil suspensions with the quinhydrone electrode in some cases give a p_H value as much as 0·8 too high. The colorimetric method is also subject to errors, but, in this case, a correction can be made covering both the "indicator" error and the influence of carbon dioxide. A correction curve is given, by the use of which the p_H of soils can be measured with an accuracy of $\pm$ 0·15 p_H unit. This curve is applicable only to soil samples examined in their natural moist condition, the proportion of soil to water being 1 : 1 by vol., and the mixture being kept for 24 hrs., with frequent stirring before filtration. It is considered that the colorimetric method is to be preferred to the quinhydrone method, and that the accuracy of the determination, though not very great, is sufficient for the purposes of most soil investigations. C. T. GIMINGHAM.

Total sulphur content of arable soil. G. BERTRAND and L. SILBERSTEIN (Compt. rend., 1927, **184**, 1388—1390). —The sulphur content of samples of arable soil has been determined by oxidising a known weight of the soil with fuming nitric acid in an open flask, thereby fixing the volatile sulphur, and finally fusing in a nickel crucible with excess of potassium carbonate and sodium carbonate. The alkali sulphate is then determined with barium chloride in the usual way. Electrical heating must be used, as fusion over a gas burner gives high results. Determinations by this method gave values for the total sulphur varying from 0·202 to 5·175 g./kg. for samples of soil from various sources. Samples of soil from the same district also showed variations, but, in general, districts having the highest sulphur content in the soil were also the most fertile; the use of sulphates as fertilisers is consequently suggested. R. BRIGHTMAN.

Laws governing the porosity of soils and their application to agriculture. W. NITZSCH (Wiss. Veröff. Siemens-Konz., 1927, **5**, 96—120).—Starting with the fact that the method of cultivating the ground influences the growth of plants, various physical factors are investigated. It is shown that there is an exact relation between the porosity, heat, and water content, and air permeability. The relation between the capillary and non-capillary components of the porosity are investigated. The water capacity of the soil as a whole is distinguished from that of its components, an increase in the proportion of fine particles increasing the capacity. Practical tests carried out from this point of view show that the results form a satisfactory basis for experiment. The ordinary methods of tilling the soil

leave much to be desired, and further work on these lines is necessary. C. J. Smithells.

Patents.

Composition for destroying weeds. H. E. Hughes, Assr. to Weed Control Co. of California (U.S.P. 1,629,819, 24.5.27. Appl., 16.9.24).—The composition comprises a mixture of arsenic trioxide, dilute hydrochloric acid, potassium permanganate, and magnesium chloride. T. S. Wheeler.

Manufacture of arsenate insecticides. H. W. Walker (U.S.P. 1,629,557, 24.5.27. Appl., 14.5.26).—Arsenic trioxide is heated in presence or absence of water with an excess of precipitated calcium carbonate, and the product, consisting of calcium carbonate coated with calcium arsenite, is heated at 650° in air to oxidise the latter salt to calcium arsenate. T. S. Wheeler.

XVII.—SUGARS; STARCHES; GUMS.

Sucrose crystallisation. I. A. Kukharenko and B. E. Krasil'shchikov (Zapiski [Russia], 1926, 4, 59—67).—A viscosimeter, constructed on the principle of a spherical body falling in a liquid, is described. The readings may be made electrically. The relation between the speed of crystallisation and the viscosity, with varying supersaturation and temperature, is linear. At constant temperature an increase in viscosity is associated with an increased speed of crystallisation; at lower temperatures the viscosity is increased and the crystallisation retarded. Chemical Abstracts.

Crystallisation of sugar solutions. J. Dědek and J. Nováček (Kolloid-Z., 1927, 42, 163—167).—A method is devised for determining the "crystallisability" of sugar solutions. A slightly undersaturated sugar solution is placed in a desiccator over calcium chloride. After evaporation has proceeded for some time, the solution is inoculated with finely-powdered sugar crystals; crystallisation takes place, and the weight of sugar crystals produced is taken as a measure of the "crystallisability" of the solution. Impure sugar solutions give smaller crystals, and the total weight of crystallised sugar is less than in the case of purified sugar solutions. Direct addition of colloids has the effect of diminishing the size of the crystals and reducing the "crystallisability." The mechanism of the process is discussed. E. S. Hedges.

Dry defecation. P. I. Filip'ev (Zapiski [Russia], 1926, 4, 72—74).—A new type of defecator is described. Sugar does not decompose during dry defecation. Chemical Abstracts.

Yeast-gum. Y. Hashitani (J. Inst. Brew., 1927, 33, 347—351).—Yeast-gum is a mannan, and is present in large quantities in yeasts of weak fermenting power. It is a white, amorphous, and hygroscopic substance which does not reduce Fehling's solution, and an aqueous solution of which shows a strong foaming power. It is unacted upon by takadiastase, kashiwagidiastase, "digestine," pancreatin, and invertase, and acetic bacteria and yeast do not grow on it. During autolysis of yeast it can pass out of the cell wall, and is found present in the beer in small quantities. Since the amount of yeast-gum in yeast is generally in inverse proportion to that of glycogen, it is considered that it does not function as a reserve substance. By depolymerisation of the gum by heating with glycerol, α-yeast-gum is obtained which is attacked by various micro-organisms, and also varies from yeast-gum in regard to its rotatory power, foaming power, and viscosity. C. Ranken.

Tragacanth and its mucilage. II. N. Evers and T. McLachlan (Pharm. J., 1927, 118, 746—747).—The deterioration of tragacanth when kept for different periods under varying conditions has been examined. Judged from the strength of the mucilage prepared from it, tragacanth undergoes considerable deterioration on keeping, this being hastened by drying and prevented by keeping in a moist atmosphere. Mucilages made in the cold after keeping for a year have a better suspending power than those made by heating (cf. B., 1924, 802). E. H. Sharples.

Action of bacilli on carbohydrates etc. Wokes and Irwin.—See XIX.

Patent.

Carbohydrate compounds (E.P. 264,261).—See V.

Cellulose from bagasse (U.S.P. 1,630,147).—See V.

XVIII.—FERMENTATION INDUSTRIES.

Original gravity [of beers]. R. H. Hopkins (J. Inst. Brew., 1927, 33, 320—336).—The figures for "degrees of gravity lost" in the Mean Brewery Table from 0·8° Spirit Indication are shown to be below the true values when applied to the determination of original gravity of fully fermented beers. The errors arise as the result of averaging into the table readings taken on partly fermented worts. Other sources of discrepancy between declared and analytically determined original gravities include variations in yeast increase, in loss of alcohol, and in the rate of fermentation among breweries. All other factors being the same, slow fermentations yield beers with higher original gravities than rapid fermentations. By utilising figures published in the Original Gravity Report of Brown and Thorpe, a revised table is drawn up giving more accurate results. C. Ranken.

Yeast reproduction in worts of varying original gravities. R. H. Hopkins (J. Inst. Brew., 1927, 33, 337—346).—Although yeast multiplication is considerably diminished in the latter stages of fermentation, yet it continues in slight degree as long as the fermentation function of the yeast is in operation, unless the oxygen supply gives out. After the cessation of multiplication, the continued increase of the yeast crops is due to the growth in size of the cells. This is especially marked in worts of high gravity. The yeast crops from worts of similar composition, up to an original gravity of 1060°, are approximately proportional to the original gravity. For gravities over 1060° the proportion does not hold. C. Ranken.

Influence of brewery water on the composition of wort and beer. V. Koudelka (Arhiv Hemiju, 1927, 1, 24—28).—The best water for the manufacture of beer is one the p_H of which is most favourable to the fermentation of wort. Waters rich in carbonates, particularly of magnesium, are unsuitable for breweries,

and cannot be improved by the addition of calcium sulphate. R. TRUSZKOWSKI.

Microscopical investigation of the dregs of wine as a means of detecting fruit wine. A. WIDMER and O. E. KALBERER (Z. Unters. Lebensm., 1927, **53**, 193—208).—An attempt to detect the adulteration of grape wine with that made from apples or pears, by reason of the starch content of the wine dregs, is described. Investigation of fresh grape-must, however, showed that starch is present in this material also, being derived chiefly from the stalks. The number of starch grains visible in the field of the microscope varies within wide limits both for fresh wine and for wine suspected of being adulterated. The shapes and modes of fission of starch grains from both grapes and fruit are very similar, so that distinction between individual grains is difficult. The presence of fruit wine can be suspected when the number of starch grains is exceptionally high and when round single grains of approximately equal size predominate. The suspicion is strengthened if, in addition to the starch, cellular tissue of pears or apples can be detected. H. J. DOWDEN.

Yeast-gum. HASHITANI—See XVII.

PATENTS.

Fermentation of sugar for the production of yeast and of alcohol. J. P. H. JANSEN (E.P. 270,770, and 271,336, 10.11.25).—Very small quantities of suitable salts, *e.g.*, phosphates, are added with pure culture yeasts to dilute solutions of sugar. (A) If the production of yeast is desired, the fermenting liquid is retained at 25—30°, and aerated by air or oxygen, the yeast being subsequently separated by centrifuging. (B) For the production of alcohol, hydrocyanic acid is added in place of the aeration. C. RANKEN.

Oxidation process, especially for use in removing iron from wine and other organic liquids. L. E. GRANDCHAMP and J. L. WOLFF (F.P. 613,922, 4.8.25).—Wine is treated with oxydases to convert ferrous compounds into ferric compounds, which are precipitated by the tannic acid in the wine and removed. L. A. COLES.

XIX.—FOODS.

Chemical composition of wheat. A. SCHHUKIN (Naukh-Agron. Zhurnal [Russia], 1926, **3**, 379—396).—The hard wheats contain more gliadin and starch than the soft, and less total nitrogen and total protein. The quantity of starch per unit of gliadin is lower in the hard wheat. The size of the kernel within the same variety has little influence on the composition. During ripening the total nitrogen decreases and the amount of gliadin, glutenin, and starch increases. In hot, dry seasons the nitrogenous substances increase and the starch decreases; in wet seasons the reverse is the case. The increase in the concentration of the soil solution influences the composition in general. The gliadin increases as the starch decreases; increase in the starch content causes a decrease of the protein. CHEMICAL ABSTRACTS.

Detection of neutralised cream. R. STROHECKER (Z. Unters. Lebensm., 1927, **53**, 221—227).—The method of Tillmans and Luckenbach (cf. B., 1925, 967) for the detection of neutralised milk has been applied to cream. Owing to the higher fat content correction is made for the greater volume of the fat phase by conversion of the hydrochloric acid titer of the iron serum (p_H 8·4—3·2) to its corresponding value for the aqueous phase (skim-milk). The acidity of the skim-milk is then read off from the curve given in the text, and from this value the acidity of the cream is calculated by multiplying by the factor $(100-f)/100$, where f is the volume of fat in 100 c.c. of cream. The difference between this value of acidity and the apparent acidity gives the extent to which the cream has been neutralised. H. J. DOWDEN.

Gelatinisation of fruit pectins. H. LÜERS and K. LOCHMÜLLER (Kolloid-Z., 1927, **42**, 154—163).—A method is described for determining the gelatinising power of fruit pectins, and the results obtained with the apparatus are given. The gelatinising power of preparations of equal pectin content rises with increase in the sugar content; conversely, keeping the sugar content constant, the gelatinising power is proportional to the concentration of the pectin. The higher the pectin content, the lower is the concentration of sugar required for gelatinisation. The gelatinising power depends also on the hydrogen-ion concentration, the optimum value being p_H 2·95—3·05. The tendency to gelatinisation falls off slowly on the acid side and rapidly on the alkaline side. Pectins containing less than 7·3% of methoxyl do not gelatinise; gelatinisation occurs most readily when the methoxyl content is 11—12%. E. S. HEDGES.

Volatile materials of food. J. KÖNIG and W. SCHREIBER (Biochem. Z., 1927, **184**, 105—124).—The volatile material obtained by heating various types of food (*e.g.*, flesh, potato, fruit, milk, egg, cauliflower, etc.) at 98—100° and removed in a stream of nitrogen is separated into carbon dioxide, hydrogen sulphide, mercaptan, phosphorus compounds, aldehydes, etc. It is found that the amount of phosphorus compounds runs parallel with the content of vitamins-*A* and -*D*. P. W. CLUTTERBUCK.

Mucous cell content of cacao husks. C. GRIEBEL and A. MIERMEISTER (Z. Unters. Lebensm., 1927, **53**, 227—233).—The determination and identification of husk in cacao products has been attempted by measuring the area covered by the mucous cells in a weighed quantity of the material (cf. B., 1926, 605). 1 pt. of husk and 9 pts. of sugar are intimately ground and 5 mg. of the powdered material are spread on a microscope slide with the aid of a few drops of benzene. The slide coating is fixed with collodion and stained with a 0·01% solution of Safranine T, whereby the mucous cells are coloured a bright orange, whilst the remaining tissue becomes deep red. The area of the cells is measured by means of a special counting microscope (*loc. cit.*). The values obtained for husks of the same native origin varied within wide limits. The same procedure was followed with fat-free cocoa powders treated with potassium bromide, and it was found that the variation in the mucous cell value followed very closely the variation in the number of scleridæ. It is possible to detect in a known cocoa powder the addition of a known

variety of husk, but such cases are rare and, in general, measurement of the mucous cell content of a cocoa powder is not so suitable as finding the scleridæ number for determining the husk content. H. J. DOWDEN.

Determination of [finely-ground] cacao husk. J. GROSSFELD (Z. Unters. Lebensm., 1927, **53**, 233—236).—It is shown that the determination of husk when very finely ground is extremely difficult by the microscopical method of Griebel and Sonntag (cf. B., 1926, 605), and that the chemical analysis is very little different from that of pure cocoa. Determination of crude fibre and nitrogenous substances by microscopical and analytical methods, in three finely-ground cocoa powders containing husk, gave ratios of nitrogenous substance to crude fibre of 1·0—1·2, which would correspond to pure cocoa husk. Determination of husk from the crude fibre content by the Grossfeld method (cf. B., 1926, 688) gave more satisfactory results. 10 g. of the material were de-fatted by ether extraction and elutriated in the manner previously described. Sedimentation was allowed to proceed for a longer period than for ordinary cocoa powder, and filtration of the residue without suction occupied several days, owing to the colloidal nature of the finely-divided husk. The deposit was dried and the crude fibre determined. The husk content, as determined from the amounts of fibre in the sediment and the original material, ranged from 12·0—15·8%. It is concluded that, although, when very finely ground, part of the husk is hydrolysed and dissolved; this loss is compensated by the tendency of the more resistant portion, which is richer in husk, to collect in the sediment. H. J. DOWDEN.

Detection and determination of vitamin-*A* and of vitamin-*D* in cod-liver oil and various food products. F. WOKES and S. G. WILLIMOTT (Pharm. J., 1927, **118**, 752—757).—Zucker's method for the detection of vitamin-*D* (by its administration producing acid fæces in rats) has been examined and found suitable for quantitative use in assay methods. Attempts to apply the method to children were unsuccessful owing to difficulty in controlling the diet. Colour tests for vitamin-*A* indicate that the characteristics of true vitamin-blue are typical absorption bands of 590 and 617 $\mu\mu$, and the fading of the blue colour with parallel formation of red or yellow. The specificity of the colour tests has also been examined quantitatively, applying certain laws of physical chemistry, and satisfactory results have been obtained during detailed studies of the destruction of the vitamin by heat and by oxidation. When cod-liver oils are examined with purified antimony trichloride, differences in the stability of the vitamin can be detected by observing difference in the colour changes. E. H. SHARPLES.

Antimony trichloride and some factors affecting its sensitivity as a reagent for vitamin-*A*. F. WOKES and J. R. BARR (Pharm. J., 1927, **118**, 758—760).—A preliminary account of the properties and factors affecting the sensitivity of antimony trichloride for use in chloroform solutions according to the method of Carr and Price (A., 1926, 870). The following results were obtained. The chloroform should be dried with anhydrous calcium chloride and be free from phosgene, chlorine, and hydrochloric acid. The antimony trichloride should be recrystallised from chloroform or other anhydrous solvent and stored in a desiccator. Solutions are preferably made in the cold, as heat may cause them to contain free chlorine which apparently accelerates the reaction between antimony trichloride and vitamin-*A*, thus diminishing the sensitivity of the reagent. This is also affected by the gradual separation of small amounts of a heavy oily liquid which is apparently a solution of chloroform in either antimony trichloride or some similar antimony compound analogous to the solution of water in phenol found in official carbolic acid solutions. The formation of this oily liquid is influenced by the presence of moisture and temperature and by exposure to air. E. H. SHARPLES.

Use of certain carbohydrates and glucosides in the differentiation of members of the *Salmonella* group of food-poisoning bacilli. F. WOKES and J. H. IRWIN (Pharm. J., 1927, **118**, 747—751).—Serological tests of twenty-one members of the *Salmonella* group of food-poisoning bacilli, together with their action on a series of twenty-four alcohols, carbohydrates, and glucosides, are described. The biochemical tests, generally, confirmed the serological classification, and for differentiation the best results were obtained with arabinose, xylose, sorbitol, mannitol, dulcitol, mannose, sucrose, and maltose. Of the glucosides, arbutin showed the greatest differentiation. Æsculin and salicin gave the same results and amygdalin and phloridzin were not acted upon. E. H. SHARPLES.

Xylene numbers [in butter analysis]. A. VAN RAALTE (Z. Unters. Lebensm., 1927, **53**, 236—244).—See B., 1926, 563.

PATENTS.

Flour improver. E. J. SULLIVAN (U.S.P. 1,630,143, 24.5.27. Appl., 26.8.26).—A mixture of monocalcium phosphate (50 pts.), ammonium chloride (25 pts.), calcium sulphate (25 pts.), and ferrous, manganese, or zinc sulphate (0·2 pt.) is used. T. S. WHEELER.

Preservation of fruit. R. DE O. MCDILL (U.S.P. 1,630,129, 24.5.27. Appl., 21.10.21).—Citrus fruit is preserved by coating it with a solution of paraffin wax in a volatile solvent, *e.g.*, petrol, and allowing the solvent to evaporate. T. S. WHEELER.

Preservation of fresh fruit, vegetables, and like food materials. A. J. H. HADDAN. From F. D. COLLINS (E.P. 271,626, 1.5.26).—The material, dehydrated to 25% of moisture, is chopped and ground, and after a short while is ground more finely and pressed into shapes. W. G. CAREY.

Method of preserving fruits. W. B. MCLAUGHLIN (U.S.P. 1,631,974, 14.6.27. Appl., 25.9.24).—The fruit is mixed with sugar and immediately milled to a fine condition before fermentation occurs, the mixture being maintained at a sufficiently low temperature to prevent fermentation until it is self-preserving. H. ROYAL-DAWSON.

Preparation of fruit for the market. W. R. BARGER, L. A. HAWKINS, and C. P. BLATZ, Assrs. to PEOPLE OF THE UNITED STATES (U.S.P. 1,632,579, 14.6.27. Appl., 25.1.26).—The fruit is washed in hot

water and subjected to a bath of dilute antiseptic for 10 min. at a temperature of 1·7°.

H. ROYAL-DAWSON.

Transference of vitamins. S. GRÖNNINGSAETER, Assr. to FISCHER HOLLINSHED Co., INC. (U.S.P. 1,629,618, 24.5.27. Appl., 2.11.26).—Cod-liver oil is heated with sodium hydroxide solution and alcohol at 80° for ½ hr., a vegetable oil and water are added, and the mixture is agitated. The greater portion of the vitamins present pass into the added oil. T. S. WHEELER.

Manufacture of powdered or dried milk products. N. C. CHRISTENSEN (E.P. 244,728, 16.11.25. Conv., 18.12.24).—See U.S.P. 1,574,233; B., 1926, 383.

Means for calculating or determining the required temperature of a liquid or semi-liquid [used in dough-making]. C. DAVIES (E.P. 272,017, 3.5.26).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of small quantities of water in alcohol. E. L. SMITH (J.C.S., 1927, 1284—1288).—Sodium or potassium is dissolved in a known volume of the alcohol, dry ethyl acetate is added, and a portion of the mixture is immediately titrated with acid; after keeping at 70° for 30 min., the free alkali liberated by the action of the water in the alcohol on the ethoxide hydrolyses the ester, and the alkali concentration for successive time intervals is determined by titration, the titration at infinite time being determined, when necessary, by extrapolation of the titration–1/time curve. The difference between the initial and limiting titrations multiplied by a factor depending on the concentration of the acid gives the percentage of water in the alcohol. The determination may be hastened and the graphical extrapolation obviated by using considerable excess of alkali metal and of ester. The accuracy is 0·01%.

S. K. TWEEDY.

Dehydration of alcohol. E. L. SMITH (J.C.S., 1927, 1288—1290).—25 g. of ethyl succinate and 7 g. of sodium are added to each litre of alcohol (containing not more than 0·5% of water); after refluxing for 2 hrs. (cf. preceding abstract) the dehydrated alcohol is distilled off through a fractionating column. The formation of ethyl ether does not take place to any measurable extent during the dehydration. The product probably contains less than 0·01% of water. S. K. TWEEDY.

Attempt to prove the use of alcohol in a case of arson. J. MAULHARDT (Arch. Pharm., 1927, **265**, 255—256).—500 g. of the uncharred wood from the fire were submitted to steam-distillation, the distillate being fractionated; 20 c.c. of first runnings, *d* 0·9972, were obtained which gave no positive test for pyridine. Acetone was identified by the iodoform and other reactions. The solution reduced ammoniacal silver nitrate solution, but ethyl alcohol could not be identified either by Berthelot's ethyl benzoate method or by the iodoform reaction after the acetone had been "fixed." The same results were obtained in control tests on a piece of partially burnt pine wood. S. COFFEY.

Modified Calvert test for ethyl phthalate. R. D. SCOTT and E. G. WILL (J. Amer. Pharm. Assoc., 1927, **16**, 417—419).—The sensitivity of this test depends on the p_H at which the red colour denoting the presence of the ethyl phthalate develops, and a technique is described to adjust it to p_H 11—12, its optimum value. The sensitivity of the test is also increased by diminishing the amount of sulphuric acid, by increasing the amount of phenol, and by using a boiling water-bath instead of a small Bunsen flame. E. A. LUNT.

Nicotine in tobacco. A. HEIDUSCHKA and F. MUTH (Pharm. Zentr., 1927, **68**, 337—345, 353—361, 369—374).—The limit of detection of nicotine is raised by the resins present in the steam-distillate from tobacco, but not sufficiently to affect the quantitative methods. Nicotine was found in the smoke from various German "nicotine-free" and "nicotine-harmless" cigarettes, and in the urine of a non-smoker after indulgence in ordinary and "nicotine-harmless" cigarettes, the chemical tests being confirmed biologically. The method of Rasmussen (A., 1916, ii, 359) is the most exact for the determination of nicotine in tobacco, whilst for comparative analyses the method of Toth as modified by Rundshagen (cf. B., 1926, 214) is recommended. The known methods for the removal of nicotine from tobacco (by evaporation, distillation, extraction, and oxidation) have been examined, and it is concluded that they do not yield a nicotine-free, aromatic tobacco which can be used in manufacture. Proposals for standards for nicotine-free tobaccos are made. B. FULLMAN.

Colour test for ergot alkaloids. N. EVERS (Pharm. J., 1927, **118**, 721—723).—The following preliminary tests based on the colour reaction with a mixture of sulphuric acid and acetic anhydride are described. For *Ext. Ergotæ Liq.*, 2 c.c. of the sample are mixed with 1 c.c. of 10% ammonia solution and shaken with 15, 10, and 5 c.c. of ether. The ether is poured off through a filter and the filtrate evaporated to dryness. The residue is dissolved in 15 c.c. of glacial acetic acid, filtered, and 4 c.c. of the filtrate are mixed with 4 c.c. of 50% sulphuric acid (by vol.). On slowly mixing the two liquids a violet-blue colour is formed which develops to a full strength in about 12 hrs., and can be compared in a tintometer against standard ergotoxine solution. To test a sample of ergot, 2 g. of the powder are shaken for 2 hrs. with 1 c.c. of 10% ammonia solution, 2 c.c. of water, and 40 c.c. of ether. The ether extract is treated as above. Results with a number of extracts are given, and all preparations showing amounts of alkaloid above 0·065% by biological test gave a well-marked colour, but the test was not always reliable in indicating inactive specimens. A negative result with the colour test always indicated an inactive preparation. Experiments with commercial samples of ergotoxine phosphate and ergotinine citrate, the latter giving a similar colour to the former, are described. E. H. SHARPLES.

Identification and determination of morphine in compound tincture of camphor. C. M. CAINES (Pharm. J., 1927, **118**, 751—752).—Arising out of the examination of a sample of compound tincture of camphor from which the whole of the meconic acid had

been precipitated as insoluble calcium and iron meconates, a method for the determination of morphine based on its colour reaction with potassium iodate in acid solution is proposed. Confirmation of the presence of morphine may be obtained by precipitation and microchemical identification, and the method is applicable to determinations of small amounts of morphine in official solutions, pills, ampullæ, and tablets.

E. H. Sharples.

Japanese peppermint oil. H. Shinosaki, T. Nagasawa, and H. Makino (Rep. Imp. Ind. Res. Inst., Osaka, 1927, 7, No. 15, 1—26).—About 10% of Hokkaido peppermint oil reacts with alkali sulphite; the reacting substance in the fraction of high b.p. is chiefly Δ^1-menthen-3-one. Menthol obtained from menthenone by reduction with nickel and hydrogen or sodium and ethyl alcohol differs in physical properties from natural menthol. Hokkaido peppermint oil usually contains twice as much menthone and sulphite-reacting substances as Okayama peppermint oil, but the latter is richer (by 10%) in menthol. Chemical Abstracts.

New constituent of lavender oil. Composition of castoreum. A. S. Pfau (Perf. & Essent. Oil Rec., 1927, 18, 205—206).—(A) Crystals deposited from a sample of lavender oil from Grasse were identified as the methyl ether of umbelliferone (7-methoxycoumarin), m.p. 117—117·5°, which has a noteworthy influence on the odour of lavender oil obtained by extraction. (B) The following compounds have been identified in castoreum: benzoic acid, benzyl alcohol, acetophenone, and *p*-ethylphenol. Other acids and phenols and a lactone having a characteristic odour are present (cf. Schimmels' Bericht, 1927).

E. H. Sharples.

Genus *Mentha*. S. B. Gordon (Perf. & Essent. Oil Rec., 1927, **18**, 219—222).—A more detailed discussion of the biochemical significance of the presence of γ-methyl-*n*-butyl alcohol in *Mentha piperita* (cf. B., 1927, 346). E. H. Sharples.

Determination of cineole. T. T. Cocking (Pharm. J., 1927, **118**, 725—727).—The author's method (B., 1921, 869 A) has been applied to the determination of cineole in commercial camphor oils, and the f.p. of mixtures of *o*-cresol with cineole, camphor, and terpene in varying proportions have been determined. Curves and tables of the results are given, and by their use the cresineol method may be used for the determination of cineole in, *e.g.*, mixtures of cineole and camphor, mixtures of terpene and cineole saturated with camphor, commercial light oil of camphor boiling below 200° or mixtures of similar composition, and crude camphor oil containing high-boiling constituents. E. H. Sharples.

Patents.

Production of substantially pure methyl alcohol. E. Audibert (E.P. 271,538, 30.1.26).—A boiling solution of pure copper nitrate or an organic copper salt (free from elements of the iron group) is treated with alkali, the precipitate washed, dried at 50°, mixed with 10—20% of the violet copper obtained by reducing black tetracupric hydroxide at 200° by Sabatier's method, and reduced with hydrogen or carbon monoxide at ordinary pressure, the temperature being kept below 200°. Alternatively, the copper nitrate or other copper salt is calcined, the oxide obtained being mixed with violet copper and reduced as above. In the presence of the catalyst thus obtained the reaction $CO + 2H_2 = CH_3{\cdot}OH$ takes place exclusively at pressures of 100 atm. upwards, the mixed gases entering the catalyst space at 160—180°, and the temperature of the space being kept at not above 400—450°. At 150 atm. upwards several hundred grams of methyl alcohol per litre of catalytic space are produced per hour. B. Fullman.

Manufacture of acetic acid and acetates. Synthetic Ammonia & Nitrates, Ltd., P. A. Smith, and H. G. Smith (E.P. 271,589, 13.3.26).—Methyl alcohol vapour is caused to react under hydrogen pressure with sodium formate at 200—300°, potassium formate or water being added to keep the formate in a molten condition. Alternatively, gases tapped from a methyl alcohol synthesis plant containing methyl alcohol vapour, hydrogen, and carbon monoxide react under pressure with caustic alkali or an alkali carbonate at a temperature not exceeding 240°, some liquid phase being continually present. Acetic acid is obtained by distilling the sodium acetate with sulphuric acid.

W. G. Carey.

Manufacture of glycol ethers. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 271,169, 22.2.26).—The condensation of alkylene oxides with monohydric alcohols in presence of sulphuric acid, alkali metal alkoxides, or alkali metal salts of lower aliphatic acids as catalysts is claimed. Examples are glycol monomethyl, monoethyl, and monobutyl ethers from ethylene oxide, and an α-chloro-βγ-dihydroxypropane monomethyl ether, b.p. 168—170°, from epichlorohydrin.

C. Hollins.

Manufacture of esters. I. G. Farbenind. A.-G. (E.P. 259,204, 27.9.26. Conv., 5.10.25).—The removal of water formed in the interaction of an alcohol and an acid is accomplished by adding a liquid such as benzene, toluene, carbon tetrachloride, hexane, etc. insoluble in water, but capable of forming with water a mixture of lower b.p. than that of either component, and also lower than that of the ternary mixture of alcohol, water, and the added liquid. By carrying out the reaction under a pressure of, *e.g.*, 12—15 atm., it is possible to distil off the water formed in the reaction, together with the added liquid, but with practically no alcohol. The added liquid is separated from the distillate and returned to the mixture. Catalysts such as sulphuric or phosphoric acid may also be added. A. Davidson.

Production of acid amides and salts. Synthetic Ammonia & Nitrates, Ltd., P. A. Smith, and H. G. Smith (E.P. 271,969, 13.3.26).—Hydrogen cyanide is passed with an alcohol vapour (methyl alcohol) over a dehydrating catalyst (alumina, thoria) at a high temperature (for methyl alcohol, 250—300°), preferably under pressure. The reaction is: $R{\cdot}OH + HCN = R{\cdot}CO{\cdot}NH_2$. The acid amide may be hydrolysed with alkali, giving a salt and ammonia. Since the latter may be used with carbon monoxide for the production of hydrogen cyanide, the process may be made cyclic.

C. Hollins.

Manufacture of unsaturated aldehydes. H. Rupe (E.P. 267,954, 17.3.27. Conv., 22.3.26; cf. A., 1926, 821).

—Acetylenic alcohols of the type, CRR′ (OH)·C:CH, are converted by treatment with an acid or an acid derivative (best with formic acid) into ethylenic aldehydes, CRR′ : CH·CHO. The acetylenic alcohols, prepared usually from acetylene and a sodio-ketone, are warmed with 85% formic acid until reaction begins. cyclo *Hexylideneacetaldehyde* (R + R′ = $[CH_2]_5$), b.p. 78—80°/10—11 mm. (*semicarbazone*, m.p. 200—205°), its 3-methyl derivative and its 2-*methyl*-5-iso*propyl* derivative, b.p. 115°/9·5 mm. (*semicarbazone*, m.p. 140·5°), ββ-dimethylacraldehyde (semicarbazone, m.p. 244°), and β-*methyl*-β-*ethylacraldehyde*, b.p. 133—134° (*semicarbazone*, m.p. 170°), are described. C. HOLLINS.

Manufacture of aralkylamines and their derivatives and intermediate products. I. G. FARBENIND. A.-G., Assees. of A.-G. FÜR ANILIN-FABR. (E.P. 249,883, 26.3.26. Conv., 30.3.25).—ω-Halogenated methylphthalimides are condensed with an aromatic compound, and the phthalyl group is removed from the product by hydrolysis. *N*-Chloromethylphthalimide is condensed in presence of zinc chloride with benzene to give benzylphthalimide, which is hydrolysed first with cold aqueous alcoholic alkali to form benzylphthalamic acid and finally with dilute acid to give benzylamine. *m*-Xylene gives 2 : 4-dimethylbenzylamine, m.p. 218—219° (hydrochloride, m.p. 210°) ; naphthalene gives N-α-*naphthylmethylphthalimide*, m.p. 175°, and (α-*naphthylmethyl*)*amine* ; phenol and anisole give, respectively, *p*-hydroxybenzylamine, m.p. 107° (with a little *o*-hydroxybenzylamine, m.p. 129°), and *p*-methoxybenzylamine (N-p-*methoxybenzylphthalimide*, m.p. 104°) ; *o*-nitrotoluene gives 3-nitro-4-methylbenzylamine. The zinc chloride catalyst may be omitted in some cases ; *e.g.*, guaiacol and chloromethylphthalimide condense at 120° to N-4-*hydroxy*-3-*methoxybenzylphthalimide*, m.p. 154°, which is hydrolysed to 4-hydroxy-3-methoxybenzylamine. C. HOLLINS.

Manufacture of aromatic aldehydes. I. G. FARBENIND. A.-G., Assees. of A.-G. FÜR ANILIN-FABR. (E.P. 250,955, 15.4.26. Conv., 15.4.25).—Aralkylamines are condensed with compounds containing reactive halogen (especially 4-chloronitrobenzene-3-sulphonic acid), the secondary amine is oxidised to an azomethine derivative, and the latter is hydrolysed by mineral acid to give an aldehyde. In conjunction with the process of E.P. 249,883 (cf. preceding abstract) this forms a convenient method for introducing an aldehyde group into an aromatic hydrocarbon or derivative, *e.g.*, vanillin from guaiacol. The preparation of 2 : 4-dimethylbenzaldehyde, anisaldehyde, vanillin, 3-chloro-4-methylbenzaldehyde, m.p. 232°, phenylacetaldehyde, α-naphthaldehyde, b.p. 134°/3 mm., and of the intermediate 4-nitro-1-aralkylaminobenzene-2-sulphonic acids and azomethines, is described. C. HOLLINS.

Preparation of di-iodotrimethylamine. L. GROUCHKINE (E.P. 272,113, 24.12.26).—*s*-Di-iodotrimethylamine, $NMe(CH_2I)_2$, is stated to be produced when αγ-di-iodohydrin (αγ-di-iodo-β-hydroxypropane), obtained by the action of potassium iodide on αγ-dichlorohydrin, is treated with excess of 20% ammonia solution and the product heated at 150° for 1 hr. The substance contains 80% of iodine, and, being free from hydriodic and iodic acids, is suitable for subcutaneous or intravenous injection. C. HOLLINS.

Manufacture of chloro-iodo-compounds of 2-aminopyridine and its derivatives. CHEM. FABR. AUF ACTIEN VORM. E. SCHERING (E.P. 264,508, 12.1.27. Conv., 15.1.26).—On addition of iodine monochloride to a solution of 2-aminopyridine in dilute hydrochloric acid an additive *compound*, probably

$$\begin{array}{l}CH\cdot CH{:}C\cdot NH_2,HCl\\ \|\qquad\quad\ \ |\\ CH\cdot CH{:}NClI\end{array}$$

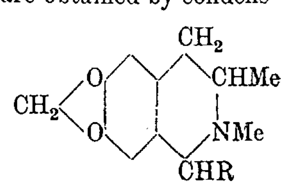
m.p. 141°, is precipitated. The corresponding 2-*acetamidopyridine chloroiodide* (m.p. 155°), and the *hydrochlorides* of 2-*ethylamino*- (m.p. 90—91°), 2-iso*propylamino*- (m.p. 84—86°), 2-iso*amylamino*- (m.p. 50—51°), 2-*diethylamino*- (m.p. 54—55°), and 2-*amino*-6-*methyl*-3-*ethyl*- (m.p. 105—106°) *pyridine chloroiodides* are similarly prepared. C. HOLLINS.

Manufacture of new compounds of bile acids. SOC. CHEM. IND. IN BASLE (GES. FÜR CHEM. IND. IN BASEL) (E.P. 257,900, 30.7.26. Conv., 2.9.25).—The salts of bile acids with asymmetric acylated aliphatic diamines are superior to the lecithin salts in emulsifying power (*e.g.*, in keeping in colloidal solution the cholesterol and calcium bilirubinate of the bile), produce an increased flow of bile, and have marked antiseptic powers. The diamine derivatives, prepared according to E.P. 203,608 and 219,304 (B., 1923, 1152 A ; 1925, 899), are mixed in molecular proportions with the bile acid in the presence or absence of a solvent, or the mixture may be triturated or fused. N-*Oleyl*-N′N′-*diethylethylenediamine dehydrocholate*, mp. 200°, N-iso*valeryl*-N′N′-*diethylethylenediamine cholate*, m.p. 90°, N-*benzoyl*-N′N′-*diethylethylenediamine cholate*, m.p. 99°, N-*stearyl*-N′N′-*diethylethylenediamine cholate*, m.p. 67°, *deoxycholate*, m.p. 58°, and *apocholate*, m.p. 132°, N-*carbomethoxy*-N′N′-*diethylethylenediamine dehydrocholate*, m.p. 199°, and N-*stearylethylenediamine dehydrocholate*, m.p. 133°, are described. C. HOLLINS.

Manufacture of mercaptobenzthiazole. W. J. KELLY, Assr. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,631,871, 7.6.27. Appl., 7.8.22).—Aniline is heated with sulphur and carbon disulphide at 150—300° under a pressure of 100—700 lb./sq. in. A. DAVIDSON.

Production of 3-pyridylhydrazine and its derivatives. DEUTSCHE GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESSLER (E.P. 259,961, 11.10.26. Conv., 17.10.25).—A 3-aminopyridine is diazotised by adding sodium nitrite solution to a solution of the base in cold concentrated hydrochloric acid, and the diazo compound is reduced with stannous chloride. The double tin salt which separates is decomposed with alkali and the hydrazine extracted with ether. 3-*Hydrazinopyridine*, m.p. 53—55°, and 2-*chloro*-5-*hydrazinopyridine*, m.p. 129—130°, are described. C. HOLLINS.

Preparation of derivatives of methylhydrastinine. CHEM. FABR. E. MERCK, Assees. of M. OBERLIN and H. MAEDER (G.P. 438,326, 2.8.25).—Intermediate products for pharmaceutical compounds are obtained by condensing methylhydrastinine in suitable media, usually in presence of alkali (sodium carbonate or sodium ethoxide) with organic compounds containing reactive hydrogen. The products described have the formula :



where R is *o*-*nitrobenzyl* (m.p. 121—122° ; by conden-

sation with o-nitrotoluene); 2-*nitro*-5-*methoxybenzyl* (m.p. 154—155°); 6-*nitro*-3 : 4-*methylenedioxybenzyl* (m.p. 178—179°); 6-*nitro*-3 : 4-*dimethoxybenzyl* (m.p. 158—160°); 1-*phenyl*-3-*methyl*-5-*hydroxy*-4-*pyrazolyl* (decomp. 130—135°; from 1-phenyl-3-methyl-5-pyrazolone); 5-*keto*-1-*phenyl*-2 : 3-*dimethyl*-4-*pyrazolenyl* (m.p. 200—201°; from antipyrin); *acetonyl* (m.p. 119—120°; from acetone); α-*carbethoxybenzyl* (m.p. 111—112°; from ethyl phenylacetate); o-*nitrobenzoyl* (decomp. 155°; from o-nitrobenzaldehyde); 6-*nitro*-3 : 4-*dimethoxybenzoyl* (decomp. 139°); 2-*nitro*-3 : 4-*dimethoxybenzoyl* (m.p. 133°); 6-*nitro*-3 : 4-*methylenedioxybenzoyl* (decomp. 143—144°); α-(6-*nitro*-3 : 4-*dimethoxyphenyl*)*allyl* (m.p. 157—158°); and α-(6-*nitro*-3 : 4-*methylenedioxyphenyl*)*allyl* (m.p. 132—133°). C. HOLLINS.

Stabilisation of molecular compounds of pyramidone and butylchloral hydrate. CHEM.-PHARM. A.-G. BAD HOMBURG (G.P. 438,984, 25.7.25).—The addition of 5—10% of hexamethylenetetramine to the molecular compound of pyramidone (4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone) with butylchloral hydrate prevents its decomposition into a brown oil without affecting its therapeutic value. The mixture is a stable, white powder. C. HOLLINS.

Preparation of a lecithin derivative. P. BERGELL (G.P. 438,328, 17.2.25).—Fresh, non-resinified lecithin is added to well-stirred anhydrous glycerol (*d* 1·26) at 90°. Solution occurs, followed by swelling to a glassy mass. This is kept for 24 hrs., finely ground in a paste mill, and worked for 1 hr. at 70—90° to form a product which is more stable and less easily hydrolysed than a simple solution of lecithin in glycerol. Mixed with concentrated sugar solutions it remains stable, and may be sterilised by heat. The product is very soluble in chloroform, from which it is incompletely precipitated by acetone, and dissolves in aqueous acetone or in ether. The lecithin is rapidly and completely racemised during the heating in glycerol, and the product is optically inactive. C. HOLLINS.

Preparation of salts of lipoid acids. BEHRINGWERKE A.-G. (G.P. 438,327, 2.11.24).—Yeasts or other micro-organisms, freed from nucleic acids by the process of G.P. 424,657 (B., 1926, 851), or the lipoid acids obtained from cells thus treated, are converted into salts by treatment with basic organic dyes, organic bases (including alkaloids), or heavy-metal salts. The products are less irritant and have less tendency to induce fever than metal or dye salts of nucleic acids. Thus, yeast free from nucleic acids is shaken in aqueous suspension with 1% fuchsine solution and kept for 12 hrs. at 15°. Excess dye is washed out and the residue of yeast cells containing the fuchsine salt of lipoid acid is dried. The yeast may be similarly treated with a quinine salt, with 3 : 3′-diamino-4 : 4′-dihydroxyarsenobenzene hydrochloride, or with mercuric chloride, to form corresponding lipoid salts. Cells freed from nucleic acid, but still containing the proteins of the bacteria nuclei, give lipoid salts mixed with salts of caryonic acid; the pharmacological properties are the same. The lipoid acids isolated by the process of G.P. 424,657 may also be used. C. HOLLINS.

Production of complex aurothiosulphate compounds. F. J. E. ANDERSEN, I. SIESBYE, and N. J. H. WEITZMANN (DANSK CHEMO-THERAPEUTISK SELSKAB VED ANDERSEN, SIESBYE, & WEITZMANN)(E.P. 261,048, 8.11.26. Conv., 7.11.25).—Aurous bromide or iodide is dissolved in a partly alcoholic solution of the bromide or iodide of an alkali metal, alkaline-earth metal, or of ammonium. The thiosulphate of the same metal is added and, after remaining for some time at 45°, sufficient alcohol is added to precipitate the aurothiosulphate. The products are of use in the treatment of tubercular diseases. A. DAVIDSON.

Manufacture of mercurimononitro-o-cresol and salts. G. W. RAIZISS, Assr. to ABBOTT LABORATORIES (U.S.P. 1,630,072, 24.5.27. Appl., 25.5.23. Cf. E.P. 236,538; B., 1925, 783).—3-Nitro-o-cresol is heated at 100° with a dilute acetic acid solution of mercuric acetate for ½ hr., treated with alkali until faintly acid, boiled, and cooled to separate crystalline 4-*acetoxymercuri*-3-*nitro*-o-*cresol*, which, on treatment with dilute sodium hydroxide solution, yields crystals of *sodium* 4-*hydroxymercuri*-3-*nitro*-o-*cresoxide*, which is of therapeutic value. T. S. WHEELER.

Preparation of a concentrated, stable solution of 3-acetamido-4-hydroxyphenylarsinic acid by the aid of its ammonium salt. ÉTABL. POULENC FRÈRES (E.P. 264,797, 3.11.26. Conv., 19.1.26).—3-Acetamido-4-hydroxyphenylarsinic acid is neutralised with 5*N*-ammonia solution; the ammonium salt is precipitated by addition of 2 vol. of alcohol and crystallised from alcohol at 60°. An aqueous solution (1 g. in 2—4 c.c.) containing a little sodium sulphite (0·1 g.) may be sterilised without deterioration. C. HOLLINS.

Manufacture of organic arsenic compounds. F. LEHNHOFF-WYLD (E.P. 249,515, 15.3.26. Conv., 17.3.25. Addn. to E.P. 232,612; B., 1925, 970).—An alcoholic solution of 1 mol. of 3 : 3′-diamino-4 : 4′-dihydroxyarsenobenzene hydrochloride is mixed with a solution, in the same alcohol, of 2 mols. of a metallic salt such as zinc chloride. The solution so obtained is added slowly to a concentrated aqueous solution of 1 mol. of the sodium salt of 3 : 3′-diamino-4 : 4′-dihydroxyarsenobenzene-*NN*′-dimethylenesulphonate. After a few hours the mixture is shaken, neutralised with alcoholic sodium hydroxide, and the product precipitated. A. DAVIDSON.

Manufacture of complex antimony compounds. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 271,940, 1.3.26).—Antimonyl derivatives of o-dihydroxybenzenes (*e.g.*, pyrocatechol, gallic acid) react with 1 mol. of a neutral salt of an aliphatic acid containing "easily migratory hydrogen atoms" (*e.g.*, malonic, lævulic, diacetylsuccinic acids) or of an aliphatic hydroxy-acid (tartaric, citric) to form new complex compounds of therapeutic value. Compounds from antimonyl pyrocatechol with sodium malonate, sodium lævulate, and sodium tartrate, and from antimonyl gallic acid with sodium malonate and sodium lævulate are described. C. HOLLINS.

Manufacture of ureides of dialkyl- or arylalkylacetic acids. F. HOFFMANN-LA ROCHE & Co. A.-G. (E.P. 264,804, 11.12.26. Conv., 20.1.26).—Substituted acetureides, which have hypnotic properties, are obtained

by prolonged action of dilute alkali upon 5:5-disubstituted barbituric acids. 5-Allyl-5-*iso*propylbarbituric acid, boiled for 100 hrs. with 5% ammonia solution or 16—17% ammonium carbonate solution or for 80 hrs. with 1% sodium hydroxide solution gives *α-isopropyl-Δ^{γ}-pentenoylcarbamide*, m.p. 194—194·5°. A mixture of sodium hydroxide, disodium hydrogen phosphate, and potassium dihydrogen phosphate may also be used for the hydrolysis, or the neutral calcium salt which may be boiled with water. 5 : 5-Dialkylbarbituric acid gives in 10 hrs. *α-allyl-Δ^{γ}-pentenoylcarbamide*, m.p. 156—157°. α-Phenylbutyrureide [α-phenylbutyrylcarbamide], m.p. 146—147°, is obtained from 5-phenyl-5-ethylbarbituric acid. C. Hollins.

Manufacture of pine extract for medical use. I. Danischewsky (E.P. 271,555, 23.2.26).—A pine needle extract completely soluble in water and containing approximately 12% of terpenes, pinenes, camphenes, and turpentine, 70% of alcohol etc., and 18% of water (slightly alkaline with sodium hydroxide) is obtained by mixing pine needle oil and alcohol or industrial methylated spirits in approximately equal quantities, and distilling at 80°. To the distillate is added 20—30% of distilled water slightly alkaline with sodium hydroxide, any oil which rises being separated. The liquid may be coloured with fluorescein etc. B. Fullman.

Manufacture of stabilised metaldehyde. E. Lüscher, Assr. to Elektrizitätswerk Lonza (U.S.P. 1,631,875, 7.6.27. Appl., 26.6.22. Conv., 3.12.21).—See E.P. 189,074; B., 1923, 44 a.

Preparation of solutions of compounds [medicaments] insoluble or sparingly soluble in water. W. Minnich, Assr. to Soc. Chem. Ind. in Basle (U.S.P. 1,632,306, 14.6.27. Appl., 10.11.24. Conv., 19.11.23).—See G.P. 420,649; B., 1926, 341.

Manufacture of acridine derivatives. I. G. Farbenind. A.-G., Assees. of H. Jensch (U.S.P. 1,629,873, 24.5.27. Appl., 3.9.21. Conv., 8.7.19).—See E.P. 176,038; B., 1922, 347 a, also A., 1922, i, 468.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic ripening nuclei. A. Steigmann (Kolloid-Z., 1927, **42**, 183—186).—A discussion of the formation and growth of ripening nuclei. In the primary process, the sensitising sulphur compound is adsorbed and then reacts, especially in the warm, to form silver sulphide. In the secondary process, silver halide is reduced by activated hydrogen obtained from the gelatin under the catalytic influence of the silver sulphide nuclei. E. S Hedges.

Patents.

Preparation of photographic emulsion and material. S. E. Sheppard and R. F. Punnett, Assrs. to Eastman Kodak Co. (U.S.P. 1,623,499, 5.4.27. Appl., 16.6.25).—An emulsion consists of a photographic silver salt suspended in a colloid, to which are added sensitising and reducing compounds upon which part at least of the sensitivity of the emulsion depends. The sensitising compound contains a bivalent atom of the sulphur group directly joined by a double linking to a single metalloid atom, to which at least another group of atoms is attached. Such compounds include thiocarbamides, selenocarbamides, etc., or the corresponding carbimides. W. Clark.

XXII.—EXPLOSIVES; MATCHES.

Patent.

Removal of tetranitromethane from [technical] trinitrotoluene. R. H. Gärtner (U.S.P. 1,632,959, 21.6.27. Appl., 18.12.24. Conv., 31.12.23).—See G.P. 416,905; B., 1926, 110.

XXIII.—SANITATION; WATER PURIFICATION.

Efficiency of chlorinating sewage tank effluents. W. V. D. Tiedeman (Eng. News-Rec., 1927, 98, 944—948).—The method of operating sewage chlorinating plants by setting a fixed minimum dosage to be used throughout the year is inefficient or uneconomical or both. Experiments, with respect to the chlorination of tank effluents from the domestic sewage of Huntington, N.Y., have shown that chlorination on a residual chlorine basis, making use of the *o*-tolidine test to control the chlorine dosage, is to be preferred. Bacteriological examinations extending over a period of 12 months show excellent results when a trace or more of free chlorine is indicated by the test. With residuals of 0·2—0·6 p.p.m., following a 10 min. contact between chlorine and sewage, reductions of 99·9% of bacteria (as evidenced by the 37° agar count) and of *B. coli* were effected. The fine solids in the tank effluents were penetrated by chlorine when a residual of 0·2 p.p.m. or more was maintained and efficient disinfection of the solids resulted. It was also observed that chlorination results in a permanent reduction in the biochemical oxygen demand of the effluent. W. T. Lockett.

Removal of iron from a ground water. H. Lührig (Gas- u. Wasserfach, 1927, **70**, 621—623).—A town water supply was found to contain iron to the extent of 2·9 mg./litre after passing through the usual purification processes. This was at first attributed to the presence of iron sulphate, but as methods for the removal of this still failed it was concluded that the iron was present in some organic form not readily oxidised by air. This was confirmed by treatment of the water with potassium permanganate (2·5—3 mg./litre), after which the iron was readily removed. A. B. Manning.

Cresol–soap systems. Jenčič.—See XII.

Patent.

Process and apparatus for electrolytically sterilising water. P. M. R. Salles (E.P. 271,721, 25.10.26).—Water is submitted to electrolytic treatment under high anodic tension in the cold in such manner that ozone is formed at an anode of small superficial area, the cathode being of much larger area. Thus the cathode may be tubular in form, constituting a duct through which water flows, whilst the anode is composed of a platinum or platinum-coated wire arranged along the axis of the duct, the distance between cathode and anode being just sufficient to permit the flow of water and disengagement of the gas evolved.

J. S. G. Thomas.

# BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 12, 1927.

I.—GENERAL; PLANT; MACHINERY.

Tentative standards, 1926. AMERICAN SOCIETY FOR TESTING MATERIALS (Separate, 1926, 1100 pp.).

CHEMICAL ABSTRACTS.

PATENTS.

Kilns. M. M. MINTER (E.P. 269,361, 18.6.26).—A series of pottery kilns are connected by ducts leading from the centre of any kiln (outlet) to the circumference, under the fire doors, of the next kiln in cyclic order. A pair of dampers are placed in each of the cyclic connexions, and cross-connexions are provided leading from between the dampers to a main induced-draught exhaust heading and to a natural-draught ring main surrounding the group of kilns. The gases from the latter may be passed through dryers for green ware. Other dampers are placed in the cross-connexions, and the usual method of operating is as follows. A kiln is fired by fuel placed in the fire doors burning in air taken through the recently burnt hot goods in the next kiln before, and the products of combustion are sent through the next kiln after, to the main induction heading. If desired, the products of combustion may pass direct from the kiln being fired to the main induction heading. The second (and possibly third) kiln before the one being fired is (or are) being finally cooled by air drawn through by natural draught to the ring main. B. M. VENABLES.

[Supply of air to] furnaces. B. LJUNGSTRÖM (E.P. 267,723, 1.4.26).—Secondary air is introduced above the fire in a furnace substantially at right angles to the movement of the bed of fuel and to the products of combustion, and is guided in at least two whirling paths, preferably rotating in opposite directions.

B. M. VENABLES.

Working shaft furnaces with the aid of furnace dust. P. HESKAMP (E.P. 269,071, 25.10.26. Conv., 6.4.26).—Blast-furnace dust is blown into the furnace shaft above the smelting zone by means of a non-oxidising gas, *e.g.*, blast-furnace gas. C. A. KING.

Refrigerating apparatus. E. L. BARNES (U.S.P. 1,625,878, 26.4.27. Appl., 22.5.25).—A refrigerating system working by the expansion and evaporation of a liquid medium under pressure is provided with a cooler on the high-pressure side for freezing and separating moisture from the liquid medium. B. M. VENABLES.

Spray cooling. B. R. SAUSEN and H. D. BINKS, Assrs. to BINKS SPRAY EQUIPMENT CO. (U.S.P. 1,626,360, 26.4.27. Appl., 19.9.21).—An upright casing open at the bottom and ventilated at the top has its lower edge dipping into an annular drip trough, the inside wall of which forms a collar which, in conjunction with a lower and smaller collar, guides an upwardly directed water spray (within the latter) across a rising current of air.

B. M. VENABLES.

Method of pulverising and apparatus therefor. W. R. WOOD (E.P. 269,319, 1.4.26).—A disintegrator comprising a shallow bowl, rotating about a vertical axis, is provided with an air draught from below the bowl past the circumference, which is deflected downwards by the walls of the chamber to the centre of the working or upper surface of the bowl, at which point the coarser material drops out of the air stream to be reground, while the air and fine material are deflected upwards again through a central exhaust pipe. The working surface of the bowl is free from ribs or other projections, but may be lined with abrasive material. B. M. VENABLES.

Pulverising machine. W. K. LIGGETT, Assr. to JEFFREY MANUF. CO. (U.S.P. 1,625,553—4, 19.4.27. Appl., 8.6.23).—(A) A disintegrator is supplied with material through a space round the shaft in an end wall. (B) A form of construction of disintegrator grating is described. B. M. VENABLES.

Roller crushing or grinding mills or machines. W. H. CAMPBELL. From J. E. HUMBERSTONE (E.P. 269,104, 14.12.26).—In a roller mill one roller is given an endways reciprocating movement by means of wedge-like devices operating behind thrust bearings, the wedges being reciprocated by eccentrics on another shaft.

B. M. VENABLES.

Tube and like mills. J. H. BENTLEY (E.P. 268,245, 3.11.26).—A tube mill with horizontal axis is constructed of a number of cylindrical sections which increase in diameter from the inlet to the outlet, the sections being separated by diaphragms each consisting of two plates with tapered holes placed together with the smallest ends of the holes in the middle or common face, so that the diameter of the apertures is not increased by wear.

B. M. VENABLES.

Machine for breaking ore and other minerals. R. HUSH (U.S.P. 1,626,535, 26.4.27. Appl., 12.2.25. Conv., 18.2.24).—In a crusher the swing jaw is directly operated (from an eccentric or crank) and the fixed jaw is adjustable by means of a wedge block formed into two pieces, one piece having a convex face and the other a corresponding concavity, to allow for variations in the angle between the jaw and crusher body when the wedge is moved. B. M. VENABLES.

Mixing machine. J. T. SIMPSON (E.P. 267,840, 12.11.26).—The machine consists essentially of an elaborated mortar mill, in which the discharge is through a central hole in the bottom of the pan. The mullers

(rollers) are adjustable, and there are usually more than one in line radially. Between each set of mullers ploughs are arranged, also in sets radially, a set being adjustable as a whole, and each plough individually as to inclination, to regulate the rate at which the material is deflected towards the central outlet. B. M. VENABLES.

Mixing machine. I. W. BENSON (E.P. 268,447, 4.1.26).—A portable concrete mixer is provided with a combined delivery chute and shovelling plate so shaped and hinged that it can be folded up within the general contour of the machine, for transport.

B. M. VENABLES.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Schramm's method for determining in the laboratory the fields of coke and by-products from coal, and its importance in coking practice. H. MÜSCHENBORN (Brennstoff-Chem., 1927, **8**, 138—140; cf. J. Gasbeleucht., 1913, **56**, 389).—Coal (15 g.) is placed in the sealed end of a glass combustion tube, with broken pieces of firebrick next to it and cotton-wool at the open end. The coal and firebrick are heated in a gas combustion furnace (the firebrick at a temperature above or below 1100°, according to large-scale conditions), the cotton-wool end of the tube is immersed in a water-bath at 90°, and the gas (freed from tar by the cotton-wool) passes through absorbing vessels containing sulphuric acid, cadmium acetate, and active charcoal to a gas receiver. The yields are in close agreement with those found in actual coking practice.

W. T. K. BRAUNHOLTZ.

"Turner" process of low-temperature carbonisation. H. ILLIES (Brennstoff-Chem., 1927, **8**, 155—156. Cf. A. W. Sanders, Iron and Coal Trades Rev., 1927, **145**, 520—522).—Coal is fed into the top of a vertical retort, superheated steam entering at the bottom and the semi-coke being continuously removed from the bottom by a worm. The volatile products leave near the upper end of the retort through a self-acting valve which opens for about 1 sec. when a given pressure is reached, and then closes for 8—20 sec. when the pressure has been released. The by-products are condensed in the usual manner, the hot cooling water from the primary cooler being returned to the steam generator. No cracking of the volatile hydrocarbons occurs, no permanent gases are formed, and the oil is almost completely free from pitch. Typical bituminous coals with 30% of volatile matter and 9—10% of ash give an average yield of about 32 gals. of oil per ton.

W. T. K. BRAUNHOLTZ.

Influence of the chemical and physical properties of brown coal on its briquetting qualities. KEGEL (Braunkohle, 1926, **25**, 389—395).—The chemical properties of brown coal (bitumen content etc.) are without appreciable influence on the briquetting qualities, which are, on the contrary, largely determined by the physical properties. To produce strong briquettes a certain water content is necessary, and also a certain composition with regard to the proportion of binder and dust. The factors determining the optimum proportion, which is different for different coals, are fully discussed.

A. B. MANNING.

Action of concentrated sulphuric acid on different types of coal. I. D. J. W. KREULEN (Brennstoff-Chem., 1927, **8**, 149—154).—Powdered samples of four different types of coal (volatile matter 41·3%, 38·4%, 22·8%, 15·4%, respectively) were warmed for different lengths of time with 96% sulphuric acid. The action is primarily oxidising, humic acid being formed and the coals losing all coking properties. The effect on each of the coals is to reduce the ash content (reckoned on the dry and ash-free basis) and calorific value, to increase the moisture content, and, in the case of the older coals, the content of volatile matter. The yield of low-temperature tar and its content of phenols is reduced, whilst its content of matter insoluble in benzene is increased. Photomicrographs of coal sections treated with concentrated sulphuric acid show that vitrain and clarain are much more readily attacked than durain and cannel coal.

W. T. K. BRAUNHOLTZ.

Ternary coal mixtures. II. D. J. W. KREULEN (Chem. Weekblad, 1927, **24**, 229—232; cf. B., 1927, 289).—The ternary mixture previously described has been further examined, and the relations between swelling and coking properties and composition expressed by means of triangular diagrams. Data and diagrams bearing on the same properties are given for other ternary mixtures. S. I. LEVY.

Cracking process. A. SACHANEN (SACHANOV) and M. TILITSCHEYEV (Petroleum, 1927, **23**, 521—525).—A wax oil of *d* 0·883 was cracked in a still of 2½ litres capacity provided with dephlegmator and condenser and with pressure regulation. The products were analysed by Egloff's method. The yield of benzine and kerosene increased with the duration of cracking up to a maximum not dependent upon the temperature. The yield of benzine was 38—39%, and of benzine and kerosene 65%, higher yields being obtainable by repeated cracking. The time to attain the maximum yield is influenced by temperature, the rapidity of cracking increasing some five times for each increase of 25° from 375° to 500°. The residue had an increased resin acid number and *d* up to 1·04. Long duration of cracking favours the production of asphaltenes and carboids. The cracking of solar oil is twice as slow as that of wax oil, and the cracking of kerosene still slower. No condensation products nor naphthenes result from the cracking of paraffin wax. Oils with high aromatic content yield the most coke. The cracked products contained 4—7% of naphthenes, 60·7—49·6% of paraffins, 14·3—19·6% of aromatics, and 7·0—25·5% of olefines. H. MOORE.

Composition and chemical constitution of lubricants (oils) and their synthesis. A. SPILKER (Petroleum, 1927, **23**, 448—451).—In synthetic research on the formation of highly viscous substances, attention was directed to the high-molecular substances obtained by addition from olefinic products with double linkings. The viscosities (Engler) at 50° of additive and condensation products of benzene homologues was: from toluene–styrene, 0·4; xylene–styrene, 0·6; ψ-cumene–styrene, 1·2; toluene–allyl alcohol, 0·9; xylene–allyl alcohol, 2·2; ψ-cumene–allyl alcohol, 16·2. The attachment of methyl groups to aromatic rings greatly increases viscosity; normal hydrocarbon chains have little viscosity, but *iso*-compounds are highly viscous. Reduc-

tion products from coal and coal tar by the Bergius process at lower temperatures and with nickel as catalyst were examined. Engler viscosities at 50° were found for the hydrogenated products of acenaphthene, 0·5; phenanthrene, 0·6; anthracene, 0·4; fluoranthrene, 1·8; pyrene, 2·3; naphthafluorene, 3·1; and chrysene, 4·5. Polyhydrides of chrysene have the characteristics of lubricants, but they are not identical with those of petroleum oils, the sp. gr. being over 1, and the hydrogen content higher. The high mol. wt. of substances derived from pitch favours the formation of highly viscous hydrogenation products. H. Moore.

Compilation of data in favour of the organic origin of petroleum. R. Potonié (Petroleum, 1927, 23, 395—399).—The author reviews the data and opinions of experts on the origin of petroleum, and the conclusions to be drawn from the presence of fossils. H. Moore.

"Saturation" of the petroleum lubricant hydrocarbons as shown by their reaction with bromine. C. F. Mabery (J. Amer. Chem. Soc., 1927, 49, 1116—1117; cf. B., 1926, 970).—A further series of brominations of various lubricants confirms the original conclusion that these hydrocarbon mixtures do not contain olefinic derivatives. F. G. Willson.

Helium in oil-well gases. Butescu and Atanasiu. —See VII.

Patents.

Washers for coal and the like. B. Norton (E.P. 263,422, 3.8.26).—Coal washers are constructed in which a layer of coal in water is pulsated by the action of air. The washers, built of sheet metal, are supported from the sides, thus allowing free access to a screw conveyor casing which removes shale from the bottom of the washer to enclosed elevator chambers at each end of the washer. The elevator chambers are produced by the extension of the sides of the washer, thus avoiding specially-cast chutes for collecting the shale from the washer screens and transferring it to the elevators. S. Pexton.

Screening apparatus for coal breakers or crushers. S. E. Page. From Pennsylvania Crusher Co. (E.P. 263,231, 24.9.25).—In a modified Bradford crusher a series of screen plates have their longitudinal edges bent at different angles so that, when built into the form of a cage by attachment to the rims of wheels carried by a central axle, the edges of the screen plates overlap and reinforce one another. S. Pexton.

Production of water-resistant fuel briquettes. Maschinenbau-A.-G. vorm. Breitfeld, Daněk & Co., and G. Plochmann (Austr. P. 104,110, 2.6.24. Conv., 13.2.24).—The material to be briquetted is air-dried and treated with the cooled vapours from the distillation of coal, wood, crude oil, oil residues, or bituminous shales, etc. in such a way that a small quantity of the vapour is absorbed by the material or precipitated thereon as a fine deposit. The resulting briquettes are highly resistant to water. A. B. Manning.

Dewatering peat. G. A. Schroter (U.S.P. 1,625,058, 19.4.27. Appl., 13.7.25).—Wet peat is coagulated into a slurry with just sufficient lime to render the mass alkaline, the liquid is then decanted and burnt lime added to the residual peat, after which it is compressed and dried in air. H. Royal-Dawson.

Production of carburetted water-gas. A. D. Little, Inc. [a], From A. D. Little. [b], From E. P. Stevenson (Can. P. 259,411—2, 21.5.24).—(a) A gas current containing the vapour of a petroleum distillate is submitted to vapour-phase cracking, the heavier residues and motor spirit are separated by condensation, and the gas, after the further removal of the hydrocarbons with three or more carbon atoms to the molecule, is mixed with water-gas. (b) Mineral oil fractions are cracked, the constituents of the gaseous product which are liquid at the normal temperature and pressure are removed, and the residual gas is submitted to a temperature higher than the cracking temperature. A. B. Manning.

Production of hydrocarbon gas. A. Gibson (Austral. P. 20,366, 3.11.24).—Air is passed through a volatile hydrocarbon in a partly-filled carburetting vessel, and the resulting gas passed through water. A. B. Manning.

Production of carbon black from natural gas. E. B. Parsons, W. D. Inskeep, and W. Hunt (U.S.P. 1,614,685, 18.1.27. Appl., 19.5.25).—Natural gas is fed through a flame and the precipitated carbon black is washed from the residual gases by means of a liquid spray (*e.g.*, water or an aqueous solution). S. Pexton.

Distillation of carbonaceous material. J. D. Zieley and F. A. Rudolf (U.S.P. 1,617,697, 15.2.27. Appl., 15.10.20).—Liquid hydrocarbons are produced by continuously distilling coal or other solid hydrocarbonaceous materials with sodium chloride. Air and steam are introduced into the base of the retort, and the volatile hydrocarbons produced are separated from the gases by condensation. S. Pexton.

Manufacture of lubricants. H. A. Montgomery (U.S.P. 1,617,455, 15.2.27. Appl., 30.8.24).—An emulsifiable oil is rendered insoluble in water by treatment with a fatty acid. Small quantities of water are then beaten into the mixture until the viscosity of the emulsion has been greatly increased. Finally, a solid lubricant is brought into suspension in the mixture by gradual addition while agitating. Lubricants so prepared are useful in metal working. S. Pexton.

Process and apparatus for distilling hydrocarbons and other liquids and fractionally condensing the vapours. R. B. Millard, E. J. Atckison, C. D. Coulter, and South Western Engineering Corp. (E.P. 264,549, 19.8.25).—The oil to be distilled is preheated by the hot residue and vapours resulting from the distillation, to about 170°, separation of vapours is permitted in a blow case in which the oil falls down a tower over suitably placed plates, the residue is heated at about 385° in a pipe still, and further evaporation takes place in a vertical tower, the evaporator being fitted with plates into which steam can be blown. The hot residue flows away to heat exchangers. The vapours from the evaporator pass into the bottom of a bubble tower, the first of a series. The vapours from the blow case pass to the bottom of the final bubble tower in the series. The bubble towers to deal with the heavy

portions of the distillate are fitted with flat perforated trays, those dealing with the lighter portions have partitions through which the vapours pass, followed by downward deflection by a bubble cap through a pool of condensate, the level of which on the partition can be adjusted. The vapours from the top of each tower pass downwards over a bank of tubes. This bank can be divided by valves into several sections, and oil on its way to the blow case, or water, flows through these sections arranged either in series, parallel, or series–parallel. Condensate and vapours, resulting from the passage of vapours over the bank of tubes, are separated in a baffled circular path, the vapours proceed to the bottom of the next bubble tower, and condensate returns to the top of the bubble tower. The temperature of the vapours on emerging from the circular path exercises a thermostatic control, short-circuiting when necessary the flow of liquid through the bank of tubes. Definite fractions are drawn off at the bottom of each bubble tower, sharp fractions being obtained. Steam can be introduced into any bubble tower as required, or the condensate from one partition may be drawn off, heated, and returned to a lower partition in that tower. The vapours from the final bubble tower are condensed as completely as possible. W. N. HOYTE.

Distillation of petroleum and similar hydrocarbons. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (E.P. 253,489, 20.4.26. Conv., 13.6.25).—The preheated oil is pumped through a pipe still where it is heated at the required temperature, and discharged into a vaporising chamber into which steam is also blown. The residue is drawn off at the bottom, and may again be heated in a pipe still with subsequent discharge to a vaporiser. The vapours from these vaporisers are led separately through a vapour separator to a rectifier, a large vessel in which further separation of vapours takes place, the separated liquid being cooled and the vapours condensed. The entire plant from the charging pump to the collecting tanks is maintained under a high vacuum. W. N. HOYTE.

Treatment of oils. W. O. SNELLING, Assr. to GASOLINE PRODUCTS CO., INC. (U.S.P. 1,624,848, 12.4.27. Appl., 5.5.15. Renewed 17.10.22).—Petroleum oils and their vapours are maintained under such conditions of heat and pressure as will produce equilibrium, and the liquid products are slowly withdrawn and replaced by fresh oil. C. O. HARVEY.

Treatment of hydrocarbons. W. M. CROSS, Assr. to GASOLINE PRODUCTS CO. (U.S.P. 1,624,889, 12.4.27. Appl., 1.6.15. Renewed 4.1.22).—The oil is heated and digested in a coil maintained in liquid communication with an enlarged chamber, and substantial equilibrium of pressure is attained between the oil and vapour in the enlarged chamber before withdrawal of the products of conversion therefrom. C. O. HARVEY.

Manufacture of gasoline. H. THOMAS, Assr. to SUN OIL CO. (U.S.P. 1,624,692, 12.4.27. Appl., 12.8.25).—Cracked hydrocarbons in liquid phase under high pressure are allowed to vaporise, and the heavier hydrocarbons condensed thereby are drawn off. A flowing stream of the vapours is partially condensed, intimately mixed with the resulting condensate, and the partially purified gasoline vapours obtained are filtered (at a controlled temperature) through a body of decolorising material wherein further partial condensation occurs. The remaining decolorised vapours are condensed. C. O. HARVEY.

Washing minerals (E.P. 268,253).—See X.

III.—TAR AND TAR PRODUCTS.

Composition and constitution of lubricants and their synthesis. SPILKER.—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

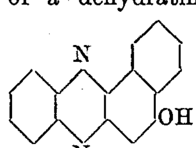
Preparing blue colours of the hydron series. E. I. ORLOV and M. CATCHOURINE (Ukraine Chem. J. Tech. Sect., 1926, 2, 65—73).—The methods of preparing colouring matters from indophenols are reviewed. 3-*p*-Benzoquinoneiminocarbazole was boiled with alcohol, and, after evaporating to dryness, treated with technical sodium sulphide (47% Na_2S, 10·7% NaHS), and the product treated with sodium hydrosulphide containing dissolved zinc. Zinc sulphide is precipitated, and the pigment goes into a solution which dyes paper violet, and from which the dyestuff is precipitated on oxidation in air. When the above iminoquinone is boiled with alcohol and sodium hexasulphide (Na_2S_6), and, after evaporating to dryness, is treated with sodium sulphide and then with sodium carbonate, a dark blue colour is produced. Since no colour is formed when the above reactions take place in sodium hydroxide solution, it is suggested that it is sodium sulphide and not alkaline sodium hydrogen sulphide that reacts with the iminoquinone. E. ROTHSTEIN.

Coal tar dyes in wines. VALENTINE.—See XVIII.

PATENTS.

Anthraquinone [acid] dyes. BRITISH DYESTUFFS CORP., LTD., and W. W. TATUM (E.P. 268,542, 19.3.26).—The acid dyes of E.P. 21,897 of 1900 (B., 1901, 1205) are found to be obtainable by the action of neutral sulphite solution on 2-halogeno-4-arylamino-1-hydroxyanthraquinones. Thus, 4-*p*-tolylamino-1-hydroxyanthraquinone-2-sulphonic acid, which dyes wool reddish-blue shades, is prepared by heating 2-chloro-4-*p*-tolylamino-1-hydroxyanthraquinone dissolved in phenol with an aqueous solution of sodium sulphite at 150—180° for 5 hrs. C. HOLLINS.

Preparation of [quinone] vat dyes. I. G. FARBENIND. A.-G., Assees. of G. KRÄNZLEIN, H. GREUNE, and H. VOLLMANN (G.P. 435,611, 28.10.24. Addn. to G.P. 433,192; cf. F.P. 593,117; B., 1926, 266).—2-Hydroxy-1 : 4-quinones are condensed, in the presence of a dehydrating agent if desired, with *p*-hydroxyphenazines, and subsequently oxidised, *e.g.*, with chlorine water. By heating 5-hydroxy-αβ-naphthaphenazine (annexed formula) with 2-hydroxy-1 : 4-naphthaquinone and acetic anhydride in nitrobenzene, there is obtained a *dye* which gives greenish-yellow shades on vegetable and animal fibres. Similar *dyes* are prepared from 2-hydroxy-1 : 4-naphthaquinone and αβ-naphthaphenazine-4 : 5-sultam or 6-chloro-5-hydroxy-αβ-naphthaphenazine. C. HOLLINS.



Manufacture of dinitro-products of perylene and its halogen derivatives. F. Bensa, Assce. of K. Stieger (E.P. 260,568, 11.10.26. Conv., 28.10.25).—Perylene or its halogen derivatives may be dinitrated by adding concentrated sulphuric acid or sulphuric–acetic acid to a hot suspension of the compound in glacial acetic acid to which has been added a concentrated aqueous solution of potassium nitrate. A yield of 96% of crude dinitroperylene is claimed, destruction by oxidation being avoided. C. Hollins.

Preparation of acid triarylmethane dyes. I. G. Farbenind. A.-G., Assees. of K. Thiess and T. Meissner (G.P. 436,830, 14.2.24).—Level-dyeing, non-bronzing, soluble acid dyes which do not resinify are obtained by oxidising a mixture of a disulphodibenzyldialkyldiaminodiarylmethane and a hydroxyalkylated arylamine. A sulphonic group may be present in the arylamine or the dye, or its leuco-derivative may be sulphonated. Disulphodibenzyldiethyldiaminodiphenylmethane, for example, is condensed with ββ-dihydroxydiethylaniline by means of dichromate and the product is salted out. C. Hollins.

Preparation of primary aromatic amines. I. G. Farbenind. A.-G., Assees. of O. Schmidt and A. Feller (G.P. 436,820, 12.1.24).—In the catalytic reduction of technical aromatic nitro-compounds, the effect of catalyst poisons is avoided by adding to the catalyst an alkaline-earth metal, aluminium, rare earths, or compounds of these. An activator, such as chromium oxide, manganese oxide, alkali, or alkali silicate, is usually also added. Thus, dry copper carbonate is ground with calcium oxide to a fine powder, pasted on pumice with sodium silicate solution, and reduced at 220—230° to form a catalyst suitable for reduction of crude nitrobenzene vapour at 200°. Technical *o*-nitrotoluene and 3-nitro-*o*-xylene may similarly be used. C. Hollins.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Patents.

Removing the dressing from fibrous vegetable material. I. G. Farbenind. A.-G., Assees. of L. Löchner and H. Korte (G.P. 434,667, 25.7.24).—The dressing is removed and adhering fats and wax are partially hydrolysed by treating the material with strongly alkaline chlorine solution; *e.g.*, starch dressing is removed by treatment for ½ hr. at 50° with a solution containing 10 g. of sodium hydroxide and 3 g. of active chlorine per litre. L. A. Coles.

Dry cleaning. A. E. Hatfield, and Achille Serre, Ltd. (E.P. 266,850, 31.12.25).—Solvents used in the dry-cleaning industry are freed from water and colloidal impurities by treatment with finely-divided porous, cellular silica of low sp. gr., *i.e.*, not more than 12 lb./cub. ft. (minimum bulk of an aqueous suspension after settling), either alone or together with decolorising carbon and reagents which are capable of flocculating suspended impurities, *e.g.*, alkali hydroxides, or carbonates, or sodium aluminate or silicate in conjunction with the equivalent quantity of an inorganic acid. The process is continuous, the treated and filtered liquor being returned to the washing machine at such a rate that the solvent therein is changed every 2—5 min. D. J. Norman.

Dry cleaning. S. G. S. Dicker. From American Dry Cleaning Co. (E.P. 266,975, 3.7.26).—The material is agitated with a low-boiling solvent the vapour of which is heavier than air and preferably non-inflammable, *e.g.*, carbon tetrachloride, in an apparatus the whole of which, including the recovery plant, is at all times in free communication with the atmosphere. The solvent left on the cleaned articles after draining is removed by a current of hot air and condensed. Specially designed plant capable of withstanding sudden changes in pressure is unnecessary, and practically the whole of the solvent is recovered. D. J. Norman.

Treatment for carroting fur. P. C. Donner (U.S.P. 1,625,458, 19.4.27. Appl., 28.4.25).—Fur is treated with an aqueous solution containing nitric and tannic acids. C. O. Harvey.

Production of artificial silk, filaments, threads, bands, ribbons, and the like. A. Eichengrün (E.P. 243,350, 17.11.25. Conv., 20.11.24).—A non-inflammable homogeneous spinning solution, which may be prepared in the cold, is obtained by dissolving acetone-soluble cellulose acetate of any degree of acetylation in a compound solvent comprising methylene chloride in admixture with alcohol, or a homologue thereof, or any cellulose acetate solvent, or solvent mixture the components of which may or may not of themselves be solvents for cellulose acetate. Mixtures containing about 30% of alcohol or 20% of acetone represent the safe limit for complete inflammability. Suitable spinning solutions are obtained by dissolving 10 pts. of acetone-soluble cellulose acetate in (*a*) 65 pts. of methylene chloride and 25 pts. of alcohol, or (*b*) 65 pts. of methylene chloride, 7·5 pts. of alcohol, and 7·5 pts. of benzene. D. J. Norman.

Spinning bath for viscose [filaments]. Spinning process for fine filaments. Production of very fine viscose silk filaments. Erste Oesterreichische Glanzstoff-Fabr. A.-G. (Austr. P. 104,237—9, [a] 21.5.20, [b] 22.3.20, and [c] 20.9.21, Addn. to Austr. P. 88,651).—(a) The bath contains sulphuric acid together with a hydroxycarboxylic acid, *e.g.*, lactic acid. (b) The bath contains a certain minimum amount of sulphuric acid depending on the thickness of the filament required. For filaments of 0—2 denier, (d_1), this minimum, (a), is 250 g./litre, and for any other denier, (d_2), it is $a\sqrt{d_1/d_2}$. (c) The bath contains sulphuric acid and ammonium sulphate together with sodium sulphate derived from the wash waters of the fibres. A. R. Powell.

Coating fibres with cellulose esters. A. É. P. Girard and M. J. P. Roumazeilles (F.P. 611,899, 16.6.25).—The fibres are steeped in a solution containing 8—10% or 10—15% of cellulose acetate, 20—30% or 20—25% of methyl acetate, acetone, or similar liquid, 15—20% or 10—15% of benzene, and 15—20% or 15—30% of a plasticising agent, for fine or coarse fibres, respectively, and are then drained and dried. The process is repeated as desired. L. A. Coles.

Treatment of cellulose. F. OLSEN and H. A. AARONSON (U.S.P. 1,615,343, 25.1.27. Appl., 9.11.25).—Cellulose is first steeped in, and later heated in an acid solution until the optimum physical degradation of the fibre with the minimum hydrolysis has taken place.

D. J. NORMAN.

Production of objects from cellulose xanthate. A. PINEL (F.P. 611,994, 2.3.26. Conv., 12.3.25).—Cellulose xanthate is treated with a solution of zinc salts, the precipitate washed, the water pressed out, and the product powdered and moulded hot with simultaneous drying. The powder can also be incorporated in plasticisers or solvents (*e.g.*, solutions of ammonium or magnesium sulphide) and worked up for plastic masses, after addition of a filler if desired.

B. FULLMAN.

Softening of artificial substances from cellulose esters. KALLE & CO. A.-G., Assees. of M. P. SCHMIDT and J. VOSS (G.P. 434,640, 5.8.23).—To cellulose esters are added ethers or thioethers of esters of glycollic acids or their homologues with higher alcohols, *e.g.*, diamyl diglycollate or thiodiglycollate, or the corresponding *cyclo*hexanol esters. B. FULLMAN.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Calcination of dolomitic limestone. H. T. BRISCOE (Proc. Indiana Acad. Sci., 1925, **35**, 133—139).—There is a relation between the rate of hydration of lime and the porosity and hardness of the stone. Lime of maximum activity is obtained by burning for the shortest time permitting complete expulsion of the carbon dioxide. Lime produced below 950° is comparatively hard and crystalline. The increased activity of lime burned at low temperatures is due to the active magnesium oxide, and to the fact that the grains have not had an opportunity to grow or partly to fuse.

CHEMICAL ABSTRACTS.

Thermal analysis of the dehydration of gypsum. R. STUMPER (Compt. rend., 1927, **184**, 970—972).—Samples of chemically pure powdered gypsum and of amorphous silica calcined at 1100° were heated at various rates (2·5—25° per min.) in an oil-bath, and the temperatures noted at intervals. The curves obtained show that the temperature of formation of the hemihydrate of gypsum increases with the rate of heating, but the temperature of formation of the anhydrite is not influenced by this factor. J. GRANT.

Production of dichromates from chromate ores with soda and lime. L. I. POPOV (Continental Met. Chem. Eng., 1926, **1**, 143—144).—Poor ores should be ground as finely as possible, the amount of lime employed should be lower than for richer ores, whilst the soda should be used in theoretical quantity. Rich ores need pass a sieve of only 2300 meshes per $cm.^2$ For ore containing 40—50% Cr_2O_3, 0·9 part of lime per 1 part of ore is preferable; for richer ores, 1·2 parts of lime are desirable. The amount of soda used may be 20% less than the theoretical. A temperature of 1100° is employed.

CHEMICAL ABSTRACTS.

Manufacture of barium chloride. H. MOLITOR (Continental Met. Chem. Eng., 1926, **1**, 139—142).—The processes employed are reviewed. A continuous extraction process is described in which the residue contains only 0·1% of barium chloride. Methods of obtaining fine and coarse crystals are described.

CHEMICAL ABSTRACTS.

Occurrence of helium in oil-well gases. D. BUTESCU and V. ATANASIU (Petroleum, 1927, **23**, 391—394).—The helium content of 40-litre samples of various well gases was ascertained. The sample was freed from carbon dioxide by potassium hydroxide and dried with calcium chloride, and the hydrocarbons, together with oxygen, were removed by cooling with liquid air. Hydrogen, nitrogen, and the rare gases were removed by adsorption with coconut charcoal at the temperature of liquid air. Remaining traces of neon and hydrogen were distinguished spectroscopically. The amount of helium was determined by admitting air to the apparatus till the helium lines appeared with the same intensity, and calculating from the known amount (0·0005%) present in air. The yellow, green, and blue lines are most readily seen. The helium content in the samples tested varied from 0·00006 to 0·0012%. H. MOORE.

PATENTS.

Manufacture of silicofluorides and products thereof. B. GEHAUF and H. W. WALKER (U.S.P. 1,617,708, 15.2.27. Appl., 14.5.26).—Silicon fluoride is made to react simultaneously with water and a basic substance, *e.g.*, sodium carbonate, to give a gelled mixture of silicofluoride and silica. This is dried and comminuted to form an insecticide.

H. ROYAL-DAWSON.

Production of lead sulphate. R. DALOZE (F.P. 611,699, 25.2.26).—Material containing lead is treated with a mixture of nitric and sulphuric acids maintained below the b.p. of nitric acid (70°), a current of air or other oxidising gas being passed through the acid.

L. A. COLES.

Manufacture of anhydrous magnesium chloride. COMP. DE PROD. CHIM. ET ELECTROMÉTALLURGIQUES ALAIS, FROGES, ET CAMARGUE (E.P. 255,042, 21.6.26. Conv., 8.7.25).—A compound (*e.g.*, fused carnallite) containing magnesium chloride in the anhydrous state is treated with anhydrous methyl or ethyl alcohol or with a mixture of these alcohols capable of dissolving the magnesium chloride without dissolving the other compounds accompanying it, separating the solution, and crystallising by evaporation. W. G. CAREY.

Manufacture of aluminium hydroxide. H. PEDERSEN, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,618,105, 15.2.27. Appl., 7.6.26. Conv., 23.5.25).—Compounds of lime and alumina from slags and similar materials are leached with a solution consisting chiefly of an alkali carbonate and a small quantity of free alkali hydroxide. The aluminium hydroxide produced is low in silica. H. ROYAL-DAWSON.

VIII.—GLASS; CERAMICS.

Microscopical examination of quartzites and lime-quartz bricks (silica bricks, Dinas bricks). J. E. HIBSCH (Feuerfest, 1926, **2**, 93—95, 113—117).—Preliminary examination of quartzites with the binocular magnifier suffices to distinguish between rock quartzite

and " cement " quartzite (amorphous). The former consists solely of quartz grains, practically uniform in size, in immediate contact; in the latter the grains are embedded in an opaque matrix or cement. Identification of all the structural ingredients is possible only by examination of thin sections under the petrological microscope, the use of which is explained in detail. Microsections of rock and cement quartzites are contrasted. Opal is never present in rock quartzites; mica flakes, however, are often present in rock quartzites, but never in cement quartzites. Rock quartzites are considered to be silicified sandstones, and the latter to have been formed by the addition of a siliceous bond to loose sands and quartz powders. Fine sands and quartz powders produced quartzites of a dense structure resembling hornstone. These quartzites are very suitable for making silica bricks; they do not require excessive grinding, and may be fired at a comparatively low temperature. Rock quartzites must be reduced to an impalpable powder, and fired at a high temperature for a long period, in order to effect the same degree of quartz conversion. In a good silica brick, at least 70% of the quartz is converted into a glass or tridymite, the remainder being visible under the microscope as small, irregular fragments embedded in a colourless glass. Cristobalite is not usually found in silica bricks until they have been in use, particularly in the Martin furnace. The microscopical examination may be supplemented by sp. gr. determinations in silica bricks in order to obtain an indication of the degree of quartz inversion.

F. Salt.

Surface deposits formed in glass furnace regenerators. H. Insley (J. Amer. Ceram. Soc., 1926, **9**, 635—638).—The deposits formed on the surfaces of two refractory bricks of high alumina content, which had been in use in glass furnace regenerators, were examined with the petrographic microscope, and were found to be composed chiefly of nephelite and carnegieite (the low- and high-temperature forms of $Na_2O,Al_2O_3,2SiO_2$) and corundum. Nephelite and carnegieite probably form only where the temperature is considerably below the glass-melting temperature. Corundum may be formed in the hotter parts of the furnace. F. Salt.

Mullite content of some American tank blocks. F. S. Thompson and H. I. Vormelker (J. Amer. Ceram. Soc., 1926, **9**, 639—640).—Samples of eight American glass tank blocks, ground to impalpable powder, were treated with pure hydrofluoric acid for 12 hrs., and the residues (mullite) weighed and analysed. Refiring the original samples increased the mullite content considerably, but the results do not justify definite conclusions regarding optimum conditions for mullite formation. The quantity of mullite formed was not proportional to the alumina content. F. Salt.

Wearing away of tank blocks. D. W. Ross (J. Amer. Ceram. Soc., 1926, **9**, 641—653).—The surfaces of used tank blocks were submitted to detailed examination. The glass dissolves exposed block material, a clay-bearing glass of higher sp. gr. than normal glass being formed. Hence, dissolution of the blocks takes place in an upward direction from horizontal joints or cracks. Where this upward dissolution occurs, the downward-facing surface of the block usually has a number of circular holes drilled vertically upward and terminating in thimble-like gas cavities. The deeper the horizontal joint or crack below the metal line in the tank the less severe is the attack. Excessively reducing atmospheres accelerate dissolution of the block material at the glass line, particularly if the batch contains excess of salt cake. F. Salt.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Manufacture of steel in India by the duplex process. B. Yaneske (Iron and Steel Inst., May, 1927, advance copy, 29 pp.).—The duplex process is a combination of the Bessemer and open-hearth methods of steel manufacture, and consists in desiliconising, and partly or almost completely decarburising molten pig iron from the blast-furnace in an acid-lined Bessemer converter, and subsequently dephosphorising the metal in the basic open-hearth furnace. The plant consists of five blast-furnaces, a hot-metal mixer of the non-active type heated either with coke-oven gas or with coal-tar, two acid-lined Bessemer converters, and two tilting open-hearth furnaces. The process, as practised by the Tata Iron and Steel Co., in India, is described in some detail. M. E. Nottage.

Action of complex saline solutions on the ferrous metals. R. Girard (Compt. rend., 1927, **184**, 818—820; cf. A., 1926, 926).—The corrosive effects of a " synthetic " sea-water containing chlorides of sodium and magnesium, and sulphates of magnesium and calcium (20, 3, 1·75, and 1·8 g./litre respectively) on steel and rusted cast iron are characterised by the nature of the products of corrosion rather than by their extent. The total effect is due to the combined effects of the individual salts, *i.e.*, calcium sulphate and sodium chloride act as precipitants in aerated solutions, the former in non-aerated solutions also, whilst corrosion is produced by the latter in non-aerated solutions, and by magnesium chloride in both types of solution.

J. Grant.

Changes in the solubility and hardness of twisted and bent iron. B. Garre (Z. anorg. Chem., 1927, **161**, 305—308).—The rate of dissolution of iron in dilute sulphuric acid increases linearly with the number of twists to which the metal has been subjected in the torsion machine, whereas the diameter of the impression made by a standard ball under a pressure of 3000 kg. decreases logarithmically under the same conditions. Twisted rods become appreciably softer after prolonged immersion in 1% sulphuric acid, and are completely annealed by heating at 900°. The solubility of iron after bending is greater than before, and subsequent annealing, although it decreases the rate of dissolution in acid, does not reduce it to that of the unbent metal.

A. R. Powell.

Wear-resistance of carburised steel *versus* cast high-manganese steel. W. J. Merten (Trans. Amer. Soc. Steel Treat., 1927, **11**, 233—244).—Tests on flexible spring seats and pads made of case-hardened low-carbon and medium-carbon steels and 14% manganese steel showed that under high pressure and sliding motion

free from shock and pounding the case-hardened steels were more resistant to wear. This holds good for all structural conditions of manganese steel, viz., as cast, as quenched, and also reheated to give a martensitic structure. The results obtained on the case-hardened steels were more consistent, due it is thought to irregular plastic flow of the manganese steel. T. H. BURNHAM.

Inclusions in light metals [and steels] and their effect on mechanical properties. H. STEUDEL (Z. Metallk., 1927, **19**, 129—137).—Examples with photomicrographs are given of the deleterious effect of slag inclusions in chromium steels and of inclusions of $FeAl_3$ in aluminium and its alloys on the tensile strength, ductility, and hardness of the metal. In rolled bars the tensile strength is greatly reduced perpendicular to the direction of rolling, but not nearly so seriously affected parallel to this direction when the inclusions are numerous; this is due to the formation of a fibrous structure in the direction of rolling, the fibres being separated from each other by elongated particles of the foreign substance. In the case of aluminium any treatment which serves to bring the impurities into solid solution or to cause their even distribution in small particles throughout the mass tends to nullify their effect on the mechanical properties. A. R. POWELL.

Relationships between the Rockwell and Brinell numbers. S. N. PETRENKO (U.S. Bur. Standards, Tech. Paper No. 334, 1927, **21**, 195—222).—Comparative Rockwell and Brinell hardness tests have been made on a number of ferrous and non-ferrous materials. The following equations are theoretically derived Brinell number $= a/(130 - \text{Rockwell ball number}) = b/(100 - \text{Rockwell cone number})^2$. The constants a and b are evaluated from the experimental results. The equations then give an error of $\pm10\%$ in determining Brinell numbers from Rockwell numbers. Similar relationships are obtained between the tensile strengths of ferrous materials and Rockwell numbers, but do not hold for non-ferrous materials. L. M. CLARK.

Tin-iron alloy in tin-plate. E. F. KOHMAN and N. H. SANBORN (Ind. Eng. Chem., 1927, **19**, 514—518). —Increase of the time of immersion of an iron plate in molten tin increases the amount of alloy-tin (insoluble in boiling sodium plumbite), but at a gradually decreasing rate. The coating tin is unaffected. Increase of alloy-tin in this way was found to be, if anything, detrimental in respect of corrosion by fruit juice. Untinned cans are perforated much less readily than tinned cans, as would be expected on the electrochemical theory of corrosion, but bulging, owing to hydrogen generation, is greater. This may be due to the higher overvoltage of tin and the effect of depolarising agents in fruit juice. Charcoal plate was found to give better service than coke plate. It is suggested that the beneficial effect of tin in protecting against corrosion is largely due to its acting as a lubricant in can manufacture. Photomicrographs of sections of tin-plate are given. C. IRWIN.

Orthotaxy and thermotaxy. F. RINNE (Z. Metallk., 1927, **19**, 162—163).—The term "orthotaxy" is applied to the structure of a metal or other substance formed of a series of long parallel crystals in columnar formation, and the term "thermotaxy" to the same structure when it is produced by heat conditions, *e.g.*, when a metal is cast in an open mould whereby, on solidification, radial columnar crystals spread inwards perpendicular to the surface of the mould. As an example of thermotaxy the structure of artificial periclase is discussed, and shown to be precisely analogous to that of cast iron or copper (cf. Tammann and Meyer, A., 1926, 783), *i.e.*, cube planes are parallel to the cooling surface and cube edges lie in the direction of heat flow. A. R. POWELL.

Tendency of metal crystals to flow. E. SCHMID (Z. Metallk., 1927, **19**, 154—157).—The elastic limit of single crystals of metals depends on the orientation of the crystal to the direction of application of the stress. Thus, in the case of a zinc crystal, the elastic limit in a direction making an angle of 60° with the hexagonal axis is 217 g./mm.², whereas in a direction at 87° to the same axis it is 1520 g./mm.²; in the first case elongation takes place by slip along a basal plane, and in the second case along a prism face. From these and other results from differently oriented crystals a solid model has been constructed in which the length of the radius vector from the centre of the model to its surface represents the magnitude of the elastic limit of that crystal, the orientation of which is given by the relative position of the radius vector to the crystal axis. An analogous flow model of a face-centred cubic crystal has also been constructed based on theoretical considerations supported by some preliminary work of Czochralski ("Moderne Metallkunde," Berlin, 1924) on aluminium crystals. A. R. POWELL.

Corrosion of metals. W. PALMAER (Tekn. Tid. [Uppl. C., Kemi], 1926, **56**, 17—21, 27—31, 35—40).— See B., 1926, 589. CHEMICAL ABSTRACTS.

PATENTS.

[Tin-silver] alloy. W. S. IRELAND, Assr. to LIPMAN REFRIGERATION Co. (U.S.P. 1,626,038, 26.4.27. Appl., 29.1.26).—The alloy consists of 95—97·5% Sn and 2·5—5% Ag. F. G. CROSSE.

Compositions for alloying molybdenum with other metals. MOLYBDENUM CORP. OF AMERICA, Assees. of E. A. LUCAS (E.P. 245,422, 21.11.25. Conv., 30.12.24). —With the object of providing a suitable form of molybdenum for addition to molten baths of other metals, *e.g.*, steel, without loss of molybdenum, a mixture of the lower molybdenum oxides and suitable fluxing agents is formed into a fused coherent mass, having a lower m.p. than the alloying metal. The fluxing agents may include lime, iron oxide, magnesium silicate or fluoride, alumina, silica. C. A. KING.

Protecting metals against oxidation at high temperatures. BRITISH THOMSON-HOUSTON CO., LTD., Assees. of F. C. KELLEY (E.P. 250,975, 19.4.26. Conv., 17.4.25).—Metals, *e.g.*, iron, nickel, molybdenum, and tungsten, are given a surface-coating of alloy which is resistant against oxidation at high temperatures, by heating the metal at 1000—1500° in contact with a mixture of equal parts of powdered chromium and aluminium oxide (or magnesium oxide) to which 10% of powdered silicon has been added. The heating process is conducted under non-oxidising conditions, *e.g.*, in an

atmosphere of hydrogen (cf. E.P. 159,102; B., 1921, 266 A). C. A. KING.

Treatment of metal baths. F. M. BECKET, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,622,078, 22.3.27. Appl., 3.9.21. Cf. U.S.P. 1,322,158 and 1,461,643; B., 1920, 30 A; 1923, 894 A).—Light deoxidising agents, *e.g.*, magnesium, are added to steel baths in the form of an alloy with lead. The distribution of the active metal in the steel is facilitated, and the fluidity of the slag is increased by the presence of lead oxide.
T. S. WHEELER.

Working up ores and metallurgical products of various kinds containing volatilisable metals. F. KRUPP GRUSONWERK A.-G., Assees. of H. STEPHANI (E.P. 252,679, 6.4.26. Conv., 26.5.25).—In the reduction of volatilisable metals in a rotary furnace, the reducing agent is raw anthracite or lignite together with other suitable substances, introduced directly into the reaction zone, *e.g.*, by blowing it in with air or gas in the same current as the main gas supply or through the furnace wall. The material to be reduced may be preheated in a separate furnace. C. A. KING

Non-oxidising annealing furnace. V. E. LANE (U.S.P. 1,624,258, 12.4.27. Appl., 15.2.26).—A muffle furnace constructed without bottom surmounts a water-filled pit in which is fitted an elevator carrying a transverse beam. A carriage mounted to travel on this beam forms the bottom of the furnace, and may be lowered and moved transversely in either direction, so that when the carriage is in position in the furnace a similar platform mounted on the other end of the trolley closes one or other of the openings into the pit. C. A. KING.

Furnaces for roasting sulphide and other ores. E. BRACQ (E.P. 248,711, 31.12.25. Conv., 9.3.25).—A furnace having a helical hearth or a number of superimposed hearths is provided with a central chamber of considerable area in which a hollow vertical column carries the scraper holders, which may be cooled by means of air or water. The interior of the annular hearths is screened so as to allow inspection of the process through openings in the central chamber. The central chamber as a whole is given an angular or helical movement and a reciprocating vertical movement in addition to the scrapers. C. A. KING.

Washing minerals by means of liquid streams. A. FRANCE (E.P. 268,253, 25.11.26).—In a mineral washing launder having single or multiple compartment separators depending from the launder, the launder may be divided by one or more longitudinal vertical partitions which may virtually extend so as to divide the compartments underneath, the latter portion being adjustable. The partition in each separating compartment may be forked at the lower end, and means are provided for regulating the discharge area. C. A. KING.

Classifying crushed ore and the like. S. H. DOLBEAR and B. L. EASTMAN, Assrs. to SELECTIVE TREATMENT Co., LTD. (U.S.P. 1,624,134, 12.4.27. Appl., 5.1.22).—Crushed material is brought into contact with a jet of water upwardly directed in a vessel containing relatively still water. Part of the solid material is then collected by centrifugal effect. C. A. KING.

Crucible [for metals]. SIEMENS-SCHUCKERTWERKE G.M.B.H. (E.P. 263,171, 16.12.26. Conv., 17.12.25).—Metals likely to be contaminated by the material of crucible walls are melted in an iron crucible having a coating of copper or nickel on its inner face, or an inner vessel of copper or nickel may be fitted into an iron crucible. C. A. KING.

Protection of metals which come into contact with lower-melting metals. SIEMENS & HALSKE A.-G., Assees. of G. MASING (G.P. 436,093, 20.3.25).—Metal articles are provided with a protective coating having substantially the same coefficient of expansion; *e.g.*, for iron vessels a coating made from lead glass and marble or calcium fluoride prevents attack in metal baths at the highest temperatures used. C. HOLLINS.

Annealing furnace. G. KÖRBER (E.P. 267,620, 21.12.25).—A furnace is provided with a rotating hearth supported on hollow rib members arranged as the radii of a regular polygon from a hollow vertical shaft; hollow stay members are also provided arranged as the sides of the polygon, and the top is formed of hollow plates. Water for cooling is passed through the whole, the hollow shaft being provided with partitions to separate inlet from outlet. The wall of the furnace is stationary, and is provided with channels within its thickness through which compressed air for combustion is passed and thus pre-heated. B. M. VENABLES.

XI.—ELECTROTECHNICS.

PATENTS.

[Electric] furnace and method of operating the same. F. W. BROOKE, Assr. to W. SWINDELL & BROTHERS (U.S.P. 1,626,263, 26.4.27. Appl., 11.10. 22). —A charge is melted in one furnace and refined in another in substantially the same length of time, whilst a predetermined atmosphere is maintained in the furnaces.
J. S. G. THOMAS.

Copper oxide electric cell. TRANSMUTOR Co., G.M.B.H. (G.P. 437,336, 27.5.25).—A zinc electrode is arranged within a copper oxide electrode consisting of two co-axial parts protected against axial displacements. The cell cover, which forms a de-aerating chamber, is pressed down upon the outer part of the copper oxide electrode by screwing an outwardly extending projection attached to the inner part of this electrode.
J. S. G. THOMAS.

Accumulator [plate]. E. SIEG (E.P. 260,244, 13.10.26. Conv., 22.10.25).—A soft-lead accumulator plate is provided with lugs or hooks of hard lead, *e.g.*, lead containing 5—11% Sb, for suspending the plate.
J. S. G. THOMAS.

[Filling lead] accumulators] with pulpy electrolyte]. F. ALETTER and L. STRASSER (E.P. 264,539, 17.1.27. Conv., 16.1.26).—Cells are filled with a dry mixture of absorbent material, *e.g.*, silica gel, and sulphuric acid of higher concentration than is necessary for operating them, and the electrolyte is diluted by adding distilled water. J. S. G. THOMAS.

Treatment of discarded lead battery plates. A. STEWART, Assr. to C. L. CONSTANT Co. (U.S.P. 1,627,351, 3.5.27. Appl., 30.9.26).—Lead battery plates are melted

together with a dry salt which converts the lead peroxide of the paste portion of the plates into a salt which is separable from the metallic portion. J. S. G. THOMAS.

Electric incandescence lamps. H. WADE. From N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 268,986, 14.5.26).—An electric incandescence lamp is provided with an auxiliary electrode coated with a getter, *e.g.*, calcium fluoride, potassium thallous chloride, which, under the influence of an electric discharge between the auxiliary electrode and the filament, generates a gas which forms a transparent compound with volatilised material of the filament, so that light absorption by the evaporated material is reduced. J. S. G. THOMAS.

Gas-filled electric incandescence lamp with filament having increased strength. D. S. GUSTIN, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,622,826, 29.3.27. Appl., 29.11.24).—The addition of diphenylamine vapour to the gas employed to fill electric lamps increases the strength and life of the filament, the diphenylamine acting as a getter. T. S. WHEELER.

Manufacture of oxide cathodes for discharge tubes. H. WADE. From N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 269,341, 30.4.26).—A core of tungsten or molybdenum is coated with platinum and subsequently with copper. The copper film is oxidised partially or completely, and coated with one or more of the alkaline-earth metals, which is oxidised whilst the reduced copper is vaporised. J. S. G. THOMAS.

Manufacture of electro-ionic discharge tubes. EDISON SWAN ELECTRIC CO., LTD. From RADIO-RÖHREN-LABORATORIUM DR. GERD NICKEL G.M.B.H. (E.P. 232,979, 29.12.25).—The anode or cold electrodes of a discharge tube are activated by electropositive metal, *e.g.*, calcium, strontium, and barium, preferably by evaporation of the oxides of these metals mixed with a little aluminium, from the incandescent cathode or an auxiliary cathode, so that when the tube is in use the particles of these metals oscillate under the influence of the electric field and produce fluorescence at the surface of the anode or cold electrodes. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 245,146, 229,622 (B., 1925, 663), 214,262 (B., 1924, 860), 208,729 (B., 1925, 391), 184,446, and 123,126 (B., 1919, 225 A).] J. S. G. THOMAS.

Target for *X*-ray tubes. H. C. RENTSCHLER and J. W. MARDEN, Assrs. to WESTINGHOUSE LAMP CO. (U.S.P. 1,625,426—7, 19.4.27. Appl., [A], 21.12.20, [B], 17.11.22. Renewed 6.12.26).—The target of an *X*-ray tube is composed of coherent metal (A) having an at. wt. between 230 and 240, (B) uranium. J. S. G. THOMAS.

XII.—FATS; OILS; WAXES.

Marine animal oils. sperm-whale oil and spermaceti. E. ANDRÉ and M. T. FRANÇOIS (Bull. Soc. Chim. biol., 1927, 9, 117—125; cf. B., 1926, 247, 987).—The fatty matter from various parts of the sperm whale has been examined, and the properties of the fats, and some of their constituents, are recorded. The fats examined are those of the head, adipose tissue, and muscle. The last-named gives slightly higher values for density, refractive index, and acid value. The saponif. values are 129, 112, and 163, and the iodine values (Hanus) 91·5, 85·0, and 123·0, respectively. The amounts of glycerol present are 1·8, 1·3, and 5·5 g. per 100 g. of fat, corresponding to the presence of approximately 18%, 13%, and 55% of glycerides in the fats, respectively. The total fatty acids represent 62·6, 60·0, and 76·5% of the fats, and those obtained give mean mol. wts. of 301, 291, and 302, and iodine values of 97·3, 87·2, and 135·0, respectively. The unsaponifiable matter is 38·0, 40·0, and 17·5% of the total fat, and the m.p., iodine values, and acetyl values are given, together with the mean mol. wt. of the alcohols present. Samples of spermaceti have been examined, and the constants and properties of the fatty acids and unsaponifiable matter determined. Glycerol is present after saponification of the spermaceti, the amount corresponding to 7% of glycerides. A. WORMALL.

Determination of free fatty acids [in cotton-seed oil]. H. B. BATTLE (J. Oil Fat Ind., 1926, 3, 160).—Minute directions for the titration with sodium hydroxide are given. CHEMICAL ABSTRACTS.

Fractionation of linseed oil at 293°. H. D. CHATAWAY (Ind. Eng. Chem., 1927, 19, 639—640).—Two batches of linseed oil of iodine number 199·5 were heated in an oil-bath at 110° for 2 hrs. in an atmosphere of carbon dioxide, followed a day later by heating at 185° for 5 hrs. at 15 mm. pressure. The following day they were heated over a bare flame at 293° ± 4°, batch *A* at atmospheric pressure and batch *B* at 15 mm. pressure, two samples being removed from each batch hourly, the samples being used for determination of the percentage soluble or insoluble in acetone by the method previously described (cf. Whitby and Chataway; B., 1926, 551). The mol. wt. of the insoluble and soluble fraction from each sample was determined by the f.p. method in benzene at 4% concentration. The mol. wt. of the acetone-insoluble fraction indicates that it is essentially colloidal, the amount of this fraction being after 7 hrs.' heating 54·8% for batch *A* and 56·1% for batch *B*. The acetone-insoluble material is viscous, and on prolonged heating at 80° sets throughout to a solid mass; this fraction appears to be responsible for the viscosity and setting power of the treated oil. The acetone-soluble material has an apparent mol. wt. greater than that of the original oil; qualitatively this fraction is of the same consistency as the original oil, and has no marked drying properties. Raw linseed oil heated at 293° without previous heating at 185° *in vacuo* does not give rise to an insoluble fraction until or unless reduced pressure be applied. H. M. LANGTON.

Analysis of cotton seed. C. H. COX (J. Oil Fat Ind., 1926, 3, 125—127).—The preparation of cotton seed for analysis is described. The available oil in the seed is calculated from that left in the cake by assuming a constant relation between the oil and ammonia content of the cake. CHEMICAL ABSTRACTS.

Determination of oil and ammonia in cotton seed. ANON. (J. Oil Fat Ind., 1926, 3, 247—248).—Concentrated hydrochloric acid (1·5—2·0 c.c.) is absorbed in the walls of a 2·5 in. flower pot, into which 50—60 g. of seed are placed; this is heated for 1 hr.

at 120—130°, and the powdered material analysed for water, ammonia, and oil (4 hrs.' extraction). The moisture content of the fresh sample is determined.

Chemical Abstracts.

Soap colour test of sulphur olive oil. H. P. Trevithick and W. H. Dickhart (J. Oil Fat Ind., 1926, **3**, 128—129).—The oil (10 g.) is saponified with sodium hydroxide (*d* 1·16), kept for 2 hrs., cooled, dissolved in 250 c.c. of alcohol, 50 c.c. then being diluted to 200 c.c., filtered, and examined (for grading) in a Lovibond tintometer. Chemical Abstracts.

Iodine number of California sardine oil. M. S. Dunn and B. S. Hollombe (Ind. Eng. Chem., 1927, **19**, 633—634).—The iodine numbers of commercial oleic acid and of five samples of commercial oil from the California sardine (*Sardinia cærulea*) freshly caught have been determined by both the Hübl and the Hanus methods. The values obtained for the oleic acid confirmed previous values. The average value for the iodine number of the sardine oil is 177·8, the values by the two methods being in very close agreement. The authors contrast these results with those of the following related species: Japanese sardine (*Clupanodon melanosticta*) 180—187; European sardine (*Clupea sardinus*), 160·9—191·7; *Clupea harengus*, 123·5—142; and *Clupea pilchardus*, 170·4—172·7. H. M. Langton.

U.S. Government master specification for grit-cake coap. (U.S. Bur. Standards, Circ. No. 130, Nov. 23, 1926. 6 pp.).—Type A, for glass and enamel, must contain 88—93% of insoluble siliceous material, ground to pass 100-mesh, not more than 5% being retained on 200-mesh, and of which 90% shall be ground felspar. Matter volatile at 105—110° must not exceed 4%, and the total alkalinity of matter insoluble in alcohol shall not exceed 1%, calculated as Na_2CO_3, nor must free alkali exceed 0·1%, calculated as NaOH. Anhydrous soda soap shall be within 1% of the difference between 100 and the materials mentioned above. Rosin, sugar, and foreign matter must not be present. Type B, for scouring and scrubbing, must answer the same requirements, except that volatile matter may be 5%, total alkalinity 3%, free alkali 0·1%, whilst 75—85% of siliceous material, mainly quartz ground to pass 100-mesh, is allowable. The usual methods of testing are detailed. W. G. Carey.

Patents.

Decomposition of fats or oils into fatty acids and glycerol. A. S. Richardson and C. V. Conley, Assrs. to Procter & Gamble Co. (U.S.P. 1,622,974, 29.3.27. Appl., 13.3.22).—Aromatic sulphonic acids of mol. wt. less than 250, *e.g.*, benzenesulphonic acid, are employed in hot aqueous solution to hydrolyse fats and oils.

T. S. Wheeler.

Production of a [hard-water] soap compound. H. H. Hanson, Assr. to Tarratine Manuf. Co., Inc. (U.S.P. 1,623,340, 5.4.27. Appl., 6.1.23).—A neutral vegetable oil soap (100 pts.) is dissolved in a mixture of water (70 pts.) and neutralised sulphonated castor oil (50 pts.), and after heating at 80° for 1 hr., glycerol (5%) and sugar (10%) are added, and the mixture is beaten at 35° until it assumes a creamy consistency.

T. S. Wheeler.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Study of the peroxide and persulphate methods for determining chromium in chrome paint pigments. E. F. Hickson (U.S. Paint Manufrs. Assoc., Nov., 1926, Circ. No. 294, 322—326).—The determination of chromium in chrome pigments by the method described in A.S.T.M. Standards, 1924, 864, gives low results. Of several contributory factors, incomplete oxidation of the chromium with sodium peroxide is the greatest source of error. The author recommends cooling the lead filtrate (rendered slightly ammoniacal) to 10—12° before adding sodium peroxide. After digestion until effervescence ceases, the solution is boiled and cooled, and a further quantity of sodium peroxide added. Digestion and subsequent boiling to eliminate hydrogen peroxide are followed by volumetric determination of the chromium with standard ferrous sulphate and potassium dichromate solutions. The application of the silver nitrate-persulphate oxidation method used in steel analysis is also suggested as an alternative to the sodium peroxide method. Chromate is determined electrometrically or volumetrically. Experimental details are fully quoted in each case, and tabulated results obtained by various analysts using these methods show satisfactory concordance. S. S. Woolf.

Tests with Hickson's "ice-cold" oxidation method for titrating chrome-green pigments. G. G. Sward (U.S. Paint Manufrs. Assoc., Nov. 1926, Circ. No. 294, 327—328; cf. preceding abstract).—Repeat analyses of a chrome-green pigment using a single oxidation with sodium peroxide at 25° gave 5·6, 8·5, 6·3, and 9·2% of lead chromate, whereas the corresponding figures when the solution was made ice-cold, and a double treatment was given, were 9·5, 9·5, 9·6, and 9·6%. S. S. Woolf.

White paint tests on zinc chloride and creosote-treated woods. H. A. Gardner (U.S. Paint Manufrs. Assoc., Sept., 1926, Circ. No. 286, 140—146).—Of 60 Southern yellow pine panels to be subsequently primed and painted, 20 were untreated, 20 were soaked in a 3% solution of zinc chloride, and 20 were soaked in creosote (the approximate absorptions per cub. ft. being ½ lb. of zinc chloride and 5 lb. of creosote, respectively). Of each group of 20 panels 10 were flat grained and the remainder edge grained. The primers used were based on zinc dust, aluminium, red lead, and nitrocellulose, followed by two coats of a fairly stiff white lead paint or of a lead-free white, ready-mixed paint (or 3 coats of paint in the absence of primer), 10 days' drying being allowed between coats in all cases. Formulæ for primers and finishing paints are given. After the application of two coats, the creosoted panels were half covered with tin foil (attached by thin varnish) in an attempt to prevent the "bleeding" that had already occurred from extending to the third coat, but after a fortnight's roof-exposure of the completed panels, these showed varying amounts of creosote stain. The remaining panels were then in good condition. Further reports on the exposure test are to be made.

S. S. Woolf.

Viscosity and mobility readings [of lacquer solutions] compared. A. W. Van Heuckeroth (U.S.

Paint Manufrs. Assoc., Nov., 1926, Circ. No. 292, 316—319).—The "mobility" times of a series of standard mineral oils of known absolute viscosity (used in filling the Gardner–Holdt viscosity standards) were determined using the mobilometer described in Circ. No. 265. On plotting "times" against "poises," a straight line relationship obtains, and factors for converting mobilometer readings into poises are derived. The viscosities of experimental nitrocellulose solutions determined in this manner show satisfactory concordance with the results obtained with the Gardner–Holdt viscosimeter.

S. S. WOOLF.

Colour number of dry pigments, and experiments on the selective adsorption of various pigments and liquids. H. A. GARDNER (U.S. Paint Manufrs. Assoc., Dec., 1926, Circ. No. 295, 329—351).—The fact that the oil in ready-mixed paints often becomes paler during long storage is explained by the adsorption of "foots" and colouring matter in the oil by pigments. The "colour number" is the number of milligrams of methylene blue adsorbed per gram of pigment when 20 g. of the pigment are mixed with 150 c.c. of the dyestuff solution (0·25 g./litre) and shaken vigorously for 2 min. every day for a week, the dye content of the supernatant liquor being determined after settling by titration with titanous chloride solution. The colour number, acid or alkali number (number of milligrams of acid or alkali to neutralise 1 g. of pigment), and oil absorption of 72 pigment samples are tabulated. The possibility of influencing the adsorption by preliminary heating, wetting with alcohol, etc. is discussed. A further series of experiments on the wetting of pigments by various liquid combinations is described, the method adopted being to introduce a small amount of pigment into a test tube containing 10 c.c. each of two immiscible liquids, *e.g.*, water and toluene, water and turpentine, water and linseed oil, etc. The behaviour of the pigment on addition, and its final distribution after shaking for a few seconds, are tabulated for 20 typical pigments and seven pairs of liquids, and it is suggested that the phenomena recorded should be capable of practical applications, *e.g.*, the incorporation of pigments in oil without the use of grinding apparatus, separation of fine from coarse pigments without recourse to elutriation, prevention of settling in lacquers, etc., on lines indicated by the author. S. S. WOOLF.

U.S. Government master specification for turpentine (gum spirits of turpentine and steam-distilled wood turpentine) (U.S. Bur. Standards, Circular No. 86, Oct. 11, 1926. 10 pp.).—The requirements for the specified types of turpentine are :—$d_{15.5}^{15.5}$ 0·860—0·875, n_D^{20} 1·465—1·478, initial b.p. 150—160°/760 mm., not less than 90% to distil below 170°/760 mm., residue after polymerisation with 38*N*-sulphuric acid not to exceed 2%, which residue must be viscous and straw or darker coloured, and of n_D^{20} not less than 1·5. Methods of sampling, testing, etc. are detailed. The specification does not cover destructively distilled wood turpentine.

S. S. WOOLF.

U.S. Government master specification for water-resisting spar varnish. (U.S. Bur. Standards, Circular No. 103, Oct. 5, 1926, 6 pp.).—One grade only of spar varnish is described, having the following characteristics :—Clear and transparent, not darker than a solution of 3 g. of potassium dichromate in 100 c.c. of pure sulphuric acid (*d* 1·84), flash point (closed-cup) not below 30°, non-volatile matter not less than 45% by wt. ; the varnish must have good working properties and normal gloss, must set to touch in not more than 5 hrs., and dry hard and tough in not more than 24 hrs., and must not dull, frost, etc. when exposed to direct draught for 5 hrs. immediately after flowing on, and allowed to harden overnight ; the dried film must withstand cold water for 18 hrs. and boiling water for 15 min. without whitening or dulling ; the varnish must be sufficiently elastic to pass a 50% kauri reduction test at 24°. Methods of sampling, testing, etc. are detailed.

S. S. WOOLF.

XIV.—INDIA-RUBBER ; GUTTA-PERCHA.

Variation in the degree of polymerisation of rubber. P. BARY and E. FLEURENT (Compt. rend., 1927, 184, 947—948).—The proportion of the more highly polymerised portion of rubber increases on long keeping in the dark at ordinary temperatures, and decreases considerably when maintained for some days at temperatures higher than normal. B. W. ANDERSON.

XV.—LEATHER ; GLUE.

Determination of the physical properties of leather. G. POVARNIN (Collegium, 1927, 125—147).—The Russian Sole-leather Commission selects 7·5 samples per 100 for testing. 90% of these yield tensile strength figures which are within 0·1 kg./mm.² of the correct result. It has been shown that it is preferable to select two strips from the shoulder of each hide, and the error is about 10% for 6 samples selected from every 100. The error is reduced to 6% by determining the strength of pieces both lengthwise and across the direction of the hide. The errors involved in sampling from different parts of the hide are discussed, but no particular method is preferred. The different layers of the leather affect the physical properties. The grain is weaker than the flesh side, which has been shown by wearing tests. The composition of the different layers varies ; the outer layers contain more ash and water-soluble matter than the inner layers. Moisture and fats affect the leather. The tensile strength is diminished by drying, and increases with increased moisture content. Elasticity curves are given for various types of leather. The effect on the tensile strength has been determined of different modes of liming for sole leather and also of swelling. These results were obtained from 1700 samples, and show that liming at temperatures above 15° gives a lower tensile strength. Cold-limed pelts gave a greater tensile strength when tanned in cold liquors than in warm. Hides which had not been limed gave the highest tensile strength if swollen with organic acids and tanned in warm liquors. Unlimed pelts swollen with sulphuric acid for 8 hrs. and tanned in warm liquors gave a higher tensile strength than those tanned in cold liquors, but longer treatment with sulphuric acid diminished the strength. D. WOODROFFE.

XVI.—AGRICULTURE.

Potassium content of plants as an indicator of available supply in soil.—J. W. Ames and R. W. Gerdel (Soil Sci., 1927, 23, 199—224).—From experiments with soils from manured plots, it is concluded that the Neubauer seedling method can furnish information as to the supply of available potassium in soils. Optimum conditions are furnished when 100 wheat seedlings are grown in 200 g. of soil diluted with 1000 g. of sand. G. W. Robinson.

Effects of some electrolytes on kaolin and the probable relations to the soil. J. R. Skeen (Soil Sci., 1927, 23, 225—242).—Data are presented showing the effect of different substances on the turbidity, volume of sediment, and sign and intensity of the charge on particles of suspensions of kaolin which had previously been purified by prolonged dialysis. From a consideration of the migration velocity of the particles and the volume of sediment obtained it is considered by the author that of the ions used (sodium, hydrogen, calcium, barium, and aluminium) only sodium and aluminium exert a flocculating effect. Hydrochloric acid is adsorbed, but the other electrolytes show negative adsorption in the sequence NaCl, $NaOH > BaCl_2 > CaCl_2 > AlCl_3$. In concentrated solutions the particle charge is reversed in the order of intensity $Al > H > Ca$, Ba. Adsorption is not considered to be a function of p_H in clays. The amount of dissolved aluminium in soil moisture depends on the amount of phosphate present as well as on p_H; a slight decrease in both may cause a big increase in soluble aluminium and iron. The formation of hardpan is discussed. G. W. Robinson.

Effect of lime and fertilisers on the potash content of soil and crop. J. G. Lipman, A. W. Blair, and A. L. Prince (Proc. Internat. Soc. Soil Sci., 1926, 2, 201—208).—In a series of parallel experiments with soils receiving various fertilisers with and without lime, the potash content of the limed series of soils and of the stems of maize grown therein was found to be lower than the corresponding figures in the unlimed series. A. G. Pollard.

Permeability of loam soils. J. Wityn (Proc. Internat. Soc. Soil Sci., 1926, 2, 209—243).—In continuous percolation experiments, the permeability of soil to water fluctuated considerably from day to day. These fluctuations are attributed to differences in the nature of the ions leached out of the soil during the operation. The presence of calcium and of carbonates in the permeating water diminished the rate of flow; sodium ions had the reverse effect. A. G. Pollard.

Determination of soil fertility. J. Stoklasa (Proc. Internat. Soc. Soil Sci., 1926, 2, 244—254).—The effect of fertilisers on the activity of soil organisms was examined by means of the rate of carbon dioxide production in soils. Nitrogenous fertilisers increased the amount of carbon dioxide produced, sodium nitrate being slightly more effective than ammonium sulphate. Superphosphate also stimulated the organisms. The effect of farmyard manure on carbon dioxide production depended not only on the quantity used, but also on the nature of the decomposable organic matter it contained. Productivity and carbon dioxide production in soils are closely parallel. Plant residues remain in soils under ordinary cultivation for a period sufficient to maintain the supply of organic matter necessary for optimum bacterial activity. A. G. Pollard.

Degree of humification of the dead covering of forest soils. A. Němec (Proc. Internat. Soc. Soil Sci., 1926, 2, 255—258).—Humified matter can be determined by boiling the sample with 6% hydrogen peroxide solution, which renders the humified material soluble, leaving a residue of unaltered fibrous material. The proportionate humification in forest soils increases with decreasing acidity. Exposure to sunlight increases humification, which is greater in forests of broad-leaved trees than of conifers. The rate of humification seems parallelled by the rate of nitrification of soils under laboratory conditions. A. G. Pollard.

Effect of potassium ions on potatoes. Effect of root activity of plants on the soil. von Brehmer (Ernährung der Pflanze, 1926, 22, 278—282; from Chem. Zentr., 1927, I, 345).—In experiments on the effect of different potassium salts on potatoes in sand–peat cultures, the best results were obtained with potassium chloride and with dipotassium hydrogen phosphate. Complete failures were obtained with potassium sulphate and potassium carbonate. The best growth corresponded with the most acid reaction (p_H 5·9). An alkaline reaction, consequent on the application of physiologically alkaline dressings, was unfavourable. The subsoil was always more acid than the top soil when the plants were well developed. When, on account of alkaline reaction, growth was inhibited, no difference in reaction between soil and subsoil was produced. G. W. Robinson.

Surface forces of soils in the neighbourhood of their hygroscopic capacity. H. S. Wolfe (Bot. Gaz., 1926, 82, 195—206; from Chem. Zentr., 1927, I, 345).—The force with which water is retained by different soil types at different moisture contents, investigated by means of vegetation experiments, is equivalent to about 10—25 atm. when abundant moisture is present. On drying, great increases occur at or about the moisture content represented by the hygroscopic capacity. For example, with a loam soil, whilst the attractive force only increased by 25 atm. between 6% and 7%, the increase at 5% was 250 atm.; at 4%, 575 atm.; and at 3%, 875 atm. G. W. Robinson.

Solubilisation of sulphur and formation of thiosulphates in a soil rich in organic nitrogen. G. Guittonneau and J. Keilling (Compt. rend., 1927, 184, 898—901).—Sulphur mixed with soil and calcium carbonate gradually forms soluble substances, among which thiosulphates are detected, more rapidly when peptone is also present (cf. A., 1926, 545).
E. W. Wignall.

Fertilisers. F. T. Shutt (Dep. Agric. Canada, Rep. Dominion Chemist, 1926, 11—18).—Sodium chloride (400—600 lb. per acre), or more markedly Malagash salt (containing 1% K_2O), slightly increased the yield of mangels, but not of turnips or oats. Gypsum and sulphur increased the yield of potatoes, but neither substance prevented scab development.
Chemical Abstracts.

Availability of phosphoric acid in slags and natural rock (Florida) phosphate. F. T. SHUTT (Dep. Agric. Canada, Rep. Dominion Chemist, 1926, 21—24).—The solubility of rock phosphate and open-hearth slag as determined by Wagner's method was 20—30% of that obtained by shaking 1 g. for 30 min. with 500 c.c. of 2% citric acid solution, or by shaking 1 g. for 5 hrs. with 500 c.c. of 1% citric acid solution. The third method gave somewhat higher results than the second with rock phosphate and Bessemer slag, but lower results with open-hearth slag alone or mixed with rock phosphate. CHEMICAL ABSTRACTS.

Properties of the colloidal soil material. M. S. ANDERSON and S. MATTSON (U.S. Dep. Agric. Bull., 1452, 1926, 1—46).—The colloidal soil materials of widely different soils vary considerably in all their properties, except density. Variations in one property usually parallel variations in other properties, possibly owing to relationship in the chemical nature, size, and structure of the particles. Variations in properties correspond with variations in the exchangeable bases and with variations in the ratio $SiO_2 : (Al_2O_3 + F_2O_3)$, but all properties are probably not chiefly governed directly by the silica ratio. Soil colloids are more lyophobe than lyophile, but are not typical of either class. CHEMICAL ABSTRACTS.

Nutritive value of pasture. II. Seasonal variations in the productivity, botanical and chemical composition, and nutritive value of pasturage on a heavy clay soil. H. E. WOODMAN, D. L. BLUNT, and J. STEWART (J. Agric. Sci., 1927, **17**, 209—263; cf. B., 1926, 506).—The results recorded in the present paper relate to pasture in a heavy soil in a season with abundant and well-distributed rainfall, and confirm the earlier conclusions as to the high nutritive value of young grass. Attention is also directed to the fact that, so far as the composition of young grass is concerned, there is no marked effect of species. Whilst the sandy pasture of the previous experiment consisted mainly of perennial rye grass and white clover, the clay pasture had creeping bent as the dominant species. The seasonal changes in mineral composition were in general agreement with the earlier results, in that the highest percentage of lime occurred in July, but the range was more restricted. The percentage of phosphate remained roughly the same. The results are discussed in connexion with the nutrition of grazing animals. G. W. ROBINSON.

Physical properties of soils. IV. Theory of capillary phenomena in soil. W. B. HAINES (J. Agric. Sci., 1927, **17**, 264—290; cf. B., 1925, 414, 1001).—The theory of the capillary behaviour of an "ideal soil" is further developed. Over a certain range of moisture there are alternative forms of water distribution. The theory is considered in relation to capillary rise and cohesion. The moisture distribution due to capillary rise can be derived from direct measurement of suction pressure. The pressure deficiency or suction necessary to draw an air–water interface into the pores of a soil characterises capillary behaviour over a considerable moisture range. The term "entry value" is applied to it. A size distribution curve for soil interstices can be derived from a complete suction curve. G. W. ROBINSON.

Biochemical processes influencing the transformation of phosphorus in podsol soils. O. K. ZIKHMAN-KEDROV (Pochvovedenie [Russia], 1926, **21**, 115—119).—The addition of lime to podsol soils stimulates the biochemical processes which liberate phosphoric acid from the organic material. The addition of chloroform, which renders the soil population inactive, prevents the production of soluble phosphate. The humic acid fraction primarily supplies the phosphorus. CHEMICAL ABSTRACTS.

High-dispersion phase of black alkali soils. A. A. SHOSHIN (Nature Agric. Arid Region U.S.S.R., [Russia], 1926, No. 1—2, 83—87).—Columnar alkali soil has a high-dispersion phase throughout its profile, increasing with the depth; it is 54 times as great as the dispersion phase of chernozem soil at a depth of 55—65 cm. The increase in dispersion runs somewhat parallel with the increase of sodium carbonate and the peptising agent. The lower horizons of the alkali soils, where the highly dispersed phase accumulated, contained an abundance of nutrient substances; it was specially rich in potassium and phosphorus. CHEMICAL ABSTRACTS.

Aluminium hydroxide and the "freezing up" of alkali soils during reclamation. W. T. MCGEORGE, J. F. BREAZEALE, and P. S. BURGESS (Science, 1926, **64**, 504—505).—Water used for leaching soils containing an excess of alkali salts often fails to percolate (the soil "freezes up") when the solute has been largely reduced, unless the irrigation water contains soluble calcium salts. When the concentration of the sodium salts approaches a minimum, the alkalinity rapidly increases owing to the formation of sodium hydroxide by progressive replacement and hydrolysis of sodium zeolite; aluminium then passes into solution as aluminate. The alkalinity is reduced by continued washing, proportionally more aluminium hydroxide being formed at the expense of the soluble aluminate. This, together with the almost complete dispersion of the clay colloids at these alkalinities, renders the soil practically impervious to water or air. A. A. ELDRIDGE.

Zeolite formation in soils. P. S. BURGESS and W. T. MCGEORGE (Science, 1926, **64**, 652—653.)—Zeolites in soils may be of very recent origin, and may even be in process of formation. The zeolite content of a soil can be increased by the addition of solutions of sodium aluminate and sodium silicate, compounds which are often present and in process of formation in black alkali soils. At strongly acid or alkaline reactions a high concentration of the component solutions is essential for the formation of the zeolitic gel, whilst in dilute solution combination occurs within a definite p_H range only; maximum precipitation occurs at p_H 5—7, whilst at p_H 3·6 the zeolite is completely soluble. The acid zeolite was synthesised from silicic acid and soluble aluminium and sodium, potassium, calcium, or magnesium salts, present in acid soils. The zeolite molecule is most stable under alkaline conditions. The formation of zeolites in alkaline soils is attributed to the presence

of sodium carbonate originating from calcium carbonate and sodium sulphate or chloride. A. A. Eldridge.

XVIII.—FERMENTATION INDUSTRIES.

Hydrogen ions in brewing processes. III. G. Hagues (J. Inst. Brew., 1927, **33**, 262—276).—During wort boiling the amount of protein matter coagulated is independent of hydrogen-ion concentrations more alkaline than p_H 4·0—5·0. On the acid side of this value less nitrogenous matter is precipitated. The amount of "total nitrogen" remaining in the wort after protein coagulation by boiling is independent of normal hop rates. The size of the coagulum particles is independent of p_H values more alkaline than approximately 5·0, but on the acid side of this value the size is very susceptible to this factor. The more acidly adjusted the original wort is in this region, the smaller is the coagulum particle in the boiled wort. Slightly larger particles are produced by aeration and agitation and by more concentrated worts. Except at p_H 4·0—4·5, the "total acidity" of a wort, titrated to absolute neutrality, changes on boiling. During boiling, alkaline substances are formed on the acid side of p_H 4·0—4·5, and on the alkaline side there is a production of acidic substances. The further removed from p_H 4·0—4·5 that the unboiled wort is, the more of either acid or alkaline substances are formed in solution. C. Ranken.

Electric charges on yeast, and hydrogen-ion concentration: their influence on attenuation and flocculation. F. Stockhausen (Woch. Brau., 1927, **44**, 121—124, 133—138).—In suspensions of clean bottom-fermentation yeast in water, which usually have a p_H value of about 6·5, the cells are positively charged, and in an electric field they travel towards the negative electrode. Owing to the mutual repulsion of the charged cells the yeast is in the so-called powdery or non-flocculating condition. If the hydrogen-ion concentration of the liquid is increased, the positive charge on the cells is reduced and disappears at p_H 3·8, at which point the cells show their maximum tendency to flocculate. At higher acidities the cells assume a small negative charge. In brewery wort the conditions are different owing to the presence of other colloidal matters, *e.g.*, proteins, which in water are negatively charged at p_H 6·5 and neutral at p_H 5·0—4·6. Yeast cells in wort may, by adsorbing negatively charged proteins, become neutral and flocculate at lower hydrogen-ion concentrations than would be the case in acidified water. This occurs at p_H 6·3 in hopped wort, at p_H 7·1 in wort boiled without hops, and at p_H 7·3 in unboiled wort. The importance of flocculation is that it causes an early deposition of the yeast, so that attenuation does not proceed so far as when the yeast is powdery and remains longer in suspension. Electrical factors have less play with top-fermentation yeasts. In aqueous suspensions the cells have only a small positive charge, and their isoelectric point is ill-defined at about p_H 4—6. The rising of the yeast to form the head in top fermentations is mainly due not to electrical causes, but to the rapid evolution of gas. J. H. Lane.

Malt extract evaporation plant. J. Weichherz (Chem.-Ztg., 1927, **51**, 273—274, 291—293).—In evaporation of malt extracts, it is found that, in the types of plant examined, a critical concentration is reached beyond which the efficiency of the system greatly diminishes. An illustrative series of measurements is given.
L. M. Clark.

Characterisation of coal-tar dyes added to wine. R. A. Valentini (Boll. Chim. Farm., 1927, **66**, 161—164).—A sample of the wine (100 c.c.), rendered slightly alkaline with barium, potassium, or sodium hydroxide, is shaken with 20 c.c. of amyl alcohol and allowed to separate into layers. If the alcohol is violet, the presence of orchil is indicated; if pink, Biebrich Scarlet or roccelline; and if green, aminoazobenzene. If the alcohol is colourless, but on acidification turns pink, fuchsine or safranine is indicated; if yellow, aminoazobenzene, chrysoidine, or chrysaniline; or if violet, methyl violet or mauveine. Dyeing tests with wool and silk then serve to fix the nature of the actual basic dyestuff present. Similar tests are given for the acid dyestuffs, and Rota's method of analysis is described. The fact that fuchsine, sulphofuchsine, and most of the azo dyestuffs cannot be detected some time after being added to wine may be explained by the formation of insoluble compounds of the dye with normal components of wine, such as tannin, or as a result of alterations or diseases suffered by dyed wines, which are usually watered wines under the influence of foreign micro-organisms. T. H. Pope.

Patents.

Apparatus for industrial culture of ferments, yeasts, microbes, etc. C. Vigreux, Assr. to Établ. Poulenc Frères (U.S.P. 1,623,896, 5.4.27. Appl., 29.2.24. Conv., 17.3.23).—An apparatus for cultivating ferments and the like comprises a cylinder containing trays, means for circulating air at a controlled temperature through the cylinder, and means for introducing culture media and cultures to the trays, without opening the cylinder. T. S. Wheeler.

Fermentation processes. J. Van Loon (F.P. 611,663, 24.2.26. Conv., 25.2.25).—Per-compounds, such as benzoyl peroxide, are added to fermenting material.
L. A. Coles.

XIX.—FOODS.

Variations in the susceptibility of the fat in dry whole milks to oxidation when stored at various temperatures and in various atmospheres. G. E. Holm, P. A. Wright, and G. R. Greenbank (J. Dairy Sci., 1927, **10**, 33—40).—The initial change is greatly modified by the enzymes present, which are largely removed by clarification. Free moisture has a retarding effect on susceptibility to oxidation; the optimum moisture content is 2—3%. Some compound containing loosely-bound oxygen causes oxidation of the fat in a vacuum. Chemical Abstracts.

Acidity of wheat and flour. H. L. Thompson (Amer. Miller, 1927, **55**, 160).—Little lactic or acetic acid appears to be present in normal flour, the acidity being due to acid phosphates and water-soluble phosphorus, but there is much acetic and lactic acid in unsound flour. Chlorine-bleached flours show an increased acidity. The acidity increases regularly with increasing

ash content. The acidity of various grades of flour is recorded. CHEMICAL ABSTRACTS.

Determination of glutenin in wheat flour. M. J. BLISH, R. C. ABBOTT, and H. PLATENIUS (Cereal Chem., 1927, **4**, 129—135).—8 g. of flour and 0·2 g. of crystallised barium hydroxide are digested for 1 hr. with 50 c.c. of water with frequent shaking, and made up to 205 c.c. with 96% methyl alcohol, the additional 5 c.c. being added to correct for the volume of the flour. After the starch has settled, the supernatant liquid is at once poured off through a cotton plug and 50 c.c. are withdrawn for Kjeldahl nitrogen determination. The difference between the total protein and that in the extract, each calculated from the nitrogen by using the factor 5·7, gives the percentage of glutenin in the flour. The results are in close agreement with those obtained by the methods of Sharp and Gortner (B., 1924, 29) and of Blish and Sandstedt (cf. Cereal Chem., 1925, **2**, 57—67). F. R. ENNOS.

Durum wheats. H. VOGEL and C. H. BAILEY (Cereal Chem., 1927, **4**, 136—149).—Durum wheat flour contains a higher average percentage of protein than flour from the vulgare wheats, but the ratio of glutenin to protein is about the same in each case (0·37). The quality constants of extracted suspensions of durum wheat flour, which average 2·87, are within the range of those of the vulgare varieties, and there is no correlation between this constant and the protein content. Extensibility tests show a lower value for doughs from the durum wheats. The two types of flour are most readily distinguished by the lower viscosity of the leached and acidulated suspensions of the durum variety. F. R. ENNOS.

Ash of hard spring wheat and its products. B. SULLIVAN and C. NEAR (Ind. Eng. Chem., 1927, **19**, 498—501).—Complete analyses of the mineral constituents of the ash derived from hard wheat by the hydrogen peroxide method and heating at 620° (cf. B., 1927, 313) show that hard and soft wheats require different treatment to give a satisfactory ash. Hard varieties require longer heating and a higher temperature, but this should not exceed 630° or fusion will occur, whilst at 600° destruction of carbonaceous matter is incomplete. Hard wheats have, in general, a higher phosphorus, a higher magnesium, and a lower potassium content than the soft grades. The phosphorus content tends to decrease with refinement of the flour, the actual compounds present being pyro- and meta-phosphates, the former predominating. The ash is accordingly always alkaline in character, the acidity often recorded being due to partial hydrolysis of the flour and consequent extraction of amino-acids. Chlorine, sodium, and sulphur compounds were found to be present in small amounts only, which is accounted for by the fact that they are present in the wheat in organic combinations which volatilise during the ashing process. In flours, the lipoid content is 20—50% higher than the fat content, as determined by ether extraction, but for middlings, bran, and germ, ether extraction gave at least as high results as the lipoid determinations. H. J. DOWDEN.

Alcohol in bread. V. SIMPSON and N. KNIGHT (Amer. Food J., 1926, **21**, 423, 443).—The percentage of alcohol in bread varied from 0·05% to 1·9%, the latter value being that for home-made bread. CHEMICAL ABSTRACTS.

Determination of starch in potatoes. G. RANKOFF (Z. Unters. Lebens., 1927, **53**, 138—146).—0·3—1·0 g. of finely-powdered potato starch (or dried potato) is shaken up with 100 c.c. of distilled water, and the resulting suspension heated on a calcium chloride bath at 110—115° for 20 min. The solution after cooling is diluted to 250 c.c. and filtered. 50 c.c. or 100 c.c. of the filtrate are treated with 60 c.c. of saturated sodium sulphate solution and sufficient iodine solution (5 g. of iodine and 10 g. of potassium iodide per litre) is added to give a yellow supernatant liquid. The precipitate is stirred and, after settling for 10—15 min., is filtered through a Gooch crucible containing asbestos covered by pumice stone, the coarser grains being in the upper layer. The precipitate, after washing with water containing sodium sulphate and iodine, is treated with 20 c.c. of sulphuric acid solution (1 : 3), and heated at 110—115° until all the iodine has been removed. The starch is then oxidised by means of boiling potassium permanganate solution. The liberated carbon dioxide is absorbed in soda lime tubes and weighed. The weight of starch is calculated by multiplying the weight of carbon dioxide produced by the factor 0·61393. H. J. DOWDEN.

Oxidising systems of fruits. W. V. CRUESS and W. Y. FONG (Fruit Products J., 1926, **6**, [3], 13—15).—The hydrogen-ion concentration markedly affects the inactivation temperatures of the oxidising systems of pears, peaches, prunes, oranges, apricots, and apples; the critical points are at about p_H 3·0 and p_H 10—11. Steaming for 3 min. at 100° destroys the peroxide, peroxydase, and catalase in halved apricots. Sulphurous acid checks the browning of apricots, pears, cherries, and peaches by inactivating the organic peroxide rather than the peroxydase; the action under normal conditions is not permanent. CHEMICAL ABSTRACTS.

Proteins. I. Amino-acids of soya bean meal. II. Amino-acids of herring meal. K. SHITA and T. YANAGIGAWA (Rep. Imp. Ind. Res. Inst., Osaka, 1926, **7**, [9], 1—16).—I. The nitrogen distribution of soya bean meal, hydrolysed by hydrochloric acid, was: amide-nitrogen 10·00, humin-nitrogen 4·83, diamino-nitrogen 26·43, monoamino-nitrogen 58·74%. Glutamic acid, aspartic acid, leucine, proline, and phenylalanine were isolated. II. Results for herring meal were: amide-nitrogen 3·18, humin-nitrogen 8·01, diamino-nitrogen 33·06, monoamino-nitrogen 55·75%. Alanine, leucine, proline, and phenylalanine were isolated. CHEMICAL ABSTRACTS.

Removal of gas from boiler-feed water. W. TÖLLER (Z. angew. Chem., 1927, **40**, 260—262).—A review of modern methods of removing oxygen from boiler feed water. Filtration through bundles of fine steel turnings appears to be the most satisfactory in that the filter can be introduced between the pump and the boiler so that there is no danger of re-absorption of oxygen during pumping. Freshly ignited wood charcoal is also an efficient deoxidiser, but is more expensive in use in that frequent heating is necessary to regenerate its absorptive properties. A. R. POWELL.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 19, 1927.

I.—GENERAL; PLANT; MACHINERY.

Micro-analytical practice in the works. A. Meixner and F. Kröcker (Mikrochem., 1927, 5, 120—133).—A brief description of the methods used by the authors in the micro-analysis of organic compounds for carbon, hydrogen, nitrogen, chlorine, sulphur, selenium, arsenic, phosphorus, vanadium, and mercury by means of the Pregl combustion apparatus, together with details of the necessary precautions to be taken to obtain accurate results. For the determination of mercury in organic compounds the substance is burnt in a current of oxygen in a small combustion tube, and the issuing gases are passed through red-hot lime and then over a small coil of gold leaf to collect the mercury. A. R. Powell.

Determination of moisture by the volatile solvent method. J. M. Jones and T. McLachlan (Analyst, 1927, 52, 383—387).—A critical examination of the volatile solvent method for determining moisture shows that, whatever solvent is used, distillation must be continued until no further water distils, and as water of hydration is removed, even with benzene, this must be allowed for. Toluene is regarded as the most generally useful solvent, and the method is particularly recommended for emulsions, including butter and oils, but not for powders etc. which do not cake. Consistent results were obtained with jams, malt extracts, etc.

D. G. Hewer.

Apparatus for gas analysis. J. N. J. Perquin (Chem. Weekblad, 1927, 24, 321—325).—An apparatus for exact analysis, arranged so that no connexions with rubber tubing are necessary to introduce either gases or absorbents, and in which all the parts are permanently joined, is described. The ordinary quartz combustion tube is replaced by one of nickel, and movements of mercury for levelling, moving the gases, etc. are effected by compressed air and water, electrically controlled. The error due to the absorption of carbon dioxide by copper oxide on cooling after a combustion, which affects the results obtained in the ordinary method, may be eliminated by sweeping out the combustion tube, while still at a red heat, with oxygen generated electrically within the apparatus; the carbon dioxide is absorbed in the burette by means of potassium hydroxide, and the oxygen by means of hyposulphite. The combustion tube is then ready for a further determination.

S. I. Levy.

Gas analysis instrument based on sound-velocity measurement. E. Griffiths (Proc. Physical Soc., 1927, 39, 300—304).—A form of gas analysis apparatus utilising stationary waves produced by a quartz crystal set into vibration piezo-electrically is described. The determination is based upon a measurement of the wave-length of the waves so produced, the instrument being more especially applicable to binary mixtures of gases, *e.g.*, air and carbon dioxide, which do not react.

J. S. G. Thomas.

Gas analysis apparatus. Frederick.—See VII.

Patents.

Mixing machines. J. Fowler & Co. (Leeds), Ltd., and C. H. Fowler (E.P. 272,151, 17.5.27).—To prevent any tendency to "balling" of the material in a rotary mixing drum, spherical projections or indentations extending inwards are provided. These are preferably equally spaced and located near the junction of the base and the side wall. H. Holmes.

Increasing the weight per unit volume of pulverulent heaped material. A. L. Mond. From I. G. Farbenind. A.-G. (E.P. 272,109, 13.12.26).—A crystalline product, especially one obtained by drying or calcination to remove water of crystallisation or other volatile constituent, is ground under pressure, *e.g.*, in an edge-runner mill, thereby compressing and compacting the crystal skeletons and, additionally in some cases, breaking up the skeletons. H. Holmes.

Filtering device. W. B. Pumphrey (U.S.P. 1,633,604, 28.6.27. Appl., 11.1.26).—Casing sections mounted on a central shaft abut on opposite sides of a ring between them, means being provided for tightening them in position. A wall of filtering material is provided within each section between a clear space within the ring and a recess within the section, to which liquid to be filtered is supplied, the filtered liquid being discharged from the recesses through a bore in the shaft.

H. Holmes.

Distillation and absorption column. C. H. Borrmann (G.P. 439,204, 12.1.22).—The separate compartments of a column in which gases or vapours are scrubbed with a counter-current of a liquid contain filling material, and are connected by nozzle tubes so narrow that the velocity of the gas or vapour passing through them prevents gas or liquid from flowing in the opposite direction. L. A. Coles.

Continuous rectification of liquids. L. Granger (F.P. 613,879, 29.7.25).—The return flow of the cooling liquid from purifying apparatus working in conjunction with a rectifier is redistilled before it is returned to the concentrator. L. A. Coles.

Clarification of liquids and recovery of the solid matter contained therein. R. J. Marx (E.P. 272,130, 3.2.27).—The clarification of liquids having solids in suspension is effected by apparatus which comprises means for the detachment of the air bubbles adhering

to the solids with a view of increasing the sp. gr. of the solids, and means, in the form of a continuous-flow settling tank, for the separation of the solids from the liquid. To remove the air bubbles, the liquid with the matter in suspension is allowed to flow, first, into a small shallow receptacle at such a rate as to overflow the rim thereof in the form of a thin film tending to form separate drops, and then to fall on to an inverted dished disc which is fixed at the surface of the liquid contained in the inlet chamber of the settling tank. The settling tank comprises a central inlet chamber of tapering form in open communication at the base with a similarly formed outer container, which is provided with an outlet at the bottom for the removal of the deposited solids and a surrounding trough at its upper part to receive the clarified effluent. W. T. LOCKETT.

Determination of [a constituent of] gas [mixtures]. R. P. MASE (U.S.P. 1,634,331, 5.7.27. Appl., 15.5.26).—A given constituent in a mixture of gases is determined by causing the mixture to flow constantly at a volumetrically uniform rate and removing the particular gas. The stream then flows through a constricted passage, and the differential pressure of the residual gas on the opposite sides of the constricted passage is indicated. W. G. CAREY.

Gas analysing apparatus. SVENSKA AKTIEBOLAGET MONO (E.P. 257,609, 24.8.26. Conv., 28.8.25).—In a gas analysis apparatus with an oscillating liquid column and with the absorption liquid used as sealing and pumping liquid, the absorption liquid is forced into a closed system of vessels partly filled with mercury. The mercury is consequently driven up until it is displaced, when the absorption liquid flows over into the measuring and absorption vessel, from which it flows back into the reservoir. Gas may thus be collected without coming into contact with the absorption liquid, which, further, is effectively renewed for each analysis in the absorption vessel. R. A. A. TAYLOR.

Gas analysis apparatus. S. S. LEVINSOHN (E.P. 272,092, 28.10.26).—The apparatus consists of a 100 c.c. measuring burette, several absorption vessels, and a capillary tube for complete combustion. The absorption vessels are made with two interconnected chambers; the gas enters the vessel, bubbles through the reagent, and is withdrawn for measurement. The measuring burette has two jackets containing water, the inner jacket or bell being connected with the capillary tube used for the combustion of the gas. A. C. MONKHOUSE.

Production of cold. I. AMUNDSEN (E.P. 266,683, 11.1.27. Conv., 27.2.26).—Activated charcoal is used to adsorb and expel, alternately, methyl alcohol or ethyl alcohol, either of which thus acts as a refrigerating agent. W. G. CAREY.

Separating materials of different specific gravities. T. M. CHANCE (Re-issue 16,674, 15.3.26, of U.S.P. 1,559,938, 7.7.22).—See B., 1926, 33.

Apparatus for charging furnaces, gas-producers, or the like. WOODALL-DUCKHAM (1920) LTD., and S. N. WELLINGTON (E.P. 272,820, 28.3.27).

Optical pyrometer. R. HASE (E.P. 272,799, 18.1.27).

Emulsions (E.P. 247,588).—See XX.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Technical and economic considerations on the better utilisation of coal with especial reference to high-pressure processes. C. KRAUCH (Stahl u. Eisen, 1927, **47**, 1118—1126).—The present position, as far as concerns the I. G. Farbenindustrie, of the problems of the economic production of hydrogen for ammonia synthesis and of the conversion of coal into liquid fuel is discussed. The cheapest process so far evolved as a solution of the first-named problem consists in using low-grade bituminous coal or lignite in a finely-divided form in a specially constructed producer in which a layer of the fuel 2 m. high is treated alternately with air and steam under just sufficient pressure to keep the particles suspended in a gas cushion so that the whole mass appears to be in a state of fluidity. The gases leave the producer at 1000°, and their heat is utilised in drying fresh charges of fuel, which is allowed to fall into the gas stream and is subsequently separated in a cyclone apparatus. The gas issuing from this passes over a catalyst which causes the carbon monoxide and the steam to react with the production of hydrogen and carbon dioxide. After removal of the latter by washing, the remaining gas consists of hydrogen and nitrogen in the correct proportions for use in the Haber process. The carbon dioxide is utilised in the ammonia-soda process or in the manufacture of ammonium sulphate from gypsum. A short historical survey of the development of the manufacture of methyl alcohol, hydrocarbons, and other organic compounds from carbon monoxide and hydrogen by the use of various catalysts, and of the direct hydrogenation of coal to obtain liquid fuels and various aliphatic and aromatic hydrocarbons by the use of catalysts and high pressures is given, followed by a discussion of the economic possibilities of products in competition with natural petroleum products. A. R. POWELL.

Carbonisation. II. Size of coal, admixture, inorganic compounds. 18TH REP. OF RES. SUB-COMM. OF GAS INVESTIGATION COMM. INST. GAS ENG. (Gas World, 1927, **86**, 622—633).—The effect of size of coal particle and admixture of coal (of different mesh), coke, calcium carbonate, iron oxide, and sodium carbonate upon the yields and properties of the products obtained from the carbonisation of a Nottinghamshire coal was investigated (cf. B., 1926, 1001). The same cronite retort was employed as in the previous experiments, but the retort temperature was reduced from 980° to 915°. The coal was graded into different sizes, 1¼ in.—¾ in., ½ in.—¼ in., and 10—30 mesh, in such a manner that each grade was of similar composition, and carbonisation tests were carried out with each grade and with mixtures of the grades. The rate of gas evolution varied slightly with the different grades, but in the case of mixed grades was reduced by as much as 14% in the early stages of carbonisation. The admixture of 10 and 20% of coke to the coal (both of 10—30-mesh) gave results which showed an increased yield of 2·7 and 1·9 therms per ton, respectively, but a decreased yield of tar per ton of 0·9 and 1·7 gals., respectively. The effect of adding 3·4% of calcium carbonate, 2·2% of iron oxide, and 3·3% of sodium carbonate to the coal (10—30 mesh) was in each

case to increase the volume and thermal value of the gas made per ton, the increase in the thermal value being greatest with sodium carbonate (12%) and least with iron oxide (2%). The calorific value of the gas was reduced owing to the presence of increased quantities of carbon monoxide, carbon dioxide, and hydrogen. The volume of the two oxides of carbon produced was in excess of that obtainable from the decomposition of the compounds added to the coal, but it is suggested that the additional hydrogen may be accounted for by the catalytic acceleration of the water-gas reaction within the coke charge. H. D. GREENWOOD.

Influence of the bitumen content of lignite dust on its explosiveness and temperature of spontaneous ignition. H. STEINBRECHER (Brennstoff-Chem., 1927, **8**, 193—195).—Removal of the bitumen by extraction raises the temperature of initial decomposition, and lowers the explosiveness of lignite dust. The nature of the bitumen also has an influence, resins imparting a greater explosiveness than waxes. The temperature of spontaneous ignition is lowered by removing the bitumen, though this effect may be masked by the opposite effect of the accompanying increase in ash content and diminution in the content of volatile matter of the dust. W. T. K. BRAUNHOLTZ.

Ternary coal mixtures. III. D. J. W. KREULEN (Chem. Weekblad, 1927, **24**, 255—258).—Further ternary mixtures of coals have been examined in the same way as before, and the factors governing the formation of coke are discussed, under the headings (1) nature of infusible residue, (2) proportions of fusible binding bitumen, and (3) properties of the bitumen; the influence of particle size is also considered (cf. B., 1927, 289). S. I. LEVY.

Retention of certain hydrocarbons by solid fuels. B. MOORE and F. S. SINNATT (Fuel, 1927, **6**, 312—318).—The rates of absorption of benzene, toluene, and xylene by powdered coal, peat, gas coke, and charcoal, at 16°, and the rates of loss of the hydrocarbon on subsequent exposure of the sample to the air have been determined. The samples were ground to pass 200-mesh, and were dried at 105°. The rate of absorption fell to a very small value after 15 hrs.' exposure to the vapour; the amounts of benzene then absorbed under the conditions of the experiment were approximately: bituminous coal 18·5%, anthracite 15·2%, gas coke 1%, peat 0·2%, animal charcoal 16·5%, wood charcoal 6·6%. Most of the absorbed vapour was readily removed, but after 480 hrs.' exposure to dry air the coal samples still retained appreciable amounts of benzene (2—3%); even considerably longer exposure to air failed to remove completely the absorbed hydrocarbon, which, however, could be recovered by passing a current of air over the fuel at 180°. Treatment of the fuels with a hydrocarbon had practically no influence on their capacity for absorbing moisture, but decreased slightly the tendency of the coals to ignite. A. B. MANNING.

Products of combustion from typical gas appliances. II. Gas fires. 17TH REP. OF RES. SUB-COMM. OF GAS INVESTIGATION COMM. INST. GAS ENG. (Gas World, 1927, **86**, 597—603).—Experiments are described to determine the amount of carbon monoxide present in the products of combustion from gas fires, the gas being burned at varying rates up to 30% in excess of the capacity of the fires and under various conditions of primary aeration. Two gas fires were selected, one being a pre-war model (1911), the other being of modern design. The results are expressed in parts of carbon monoxide per 10,000 parts of gas burned. With the modern fire and normal aeration (air–gas ratio 1·48) the carbon monoxide increases from 15 pts. at half normal gas rate to 30 pts. at the normal rate; on increasing the air–gas ratio to 1·90 the production of carbon monoxide is reduced to 16 pts. at normal gas rate. At low aeration (ratio 1·38) the older fire gives 16 pts. of carbon monoxide at half gas rate and 235 pts. at normal rate, whilst on increasing the aeration ratio to 1·87 the production of carbon monoxide is diminished to 7 and 80 pts. respectively. The smaller carbon monoxide production in the case of the modern fire is attributed to greater uniformity in the quality and rate of discharge of the air–gas mixture. Although the concentration of carbon monoxide in the flue gases is usually below 1 pt. per 10,000, this is regarded as sufficient to justify the provision of an efficient flue. H. D. GREENWOOD.

Equilibrium conditions in the formation of hydrocarbons and alcohols from water-gas. D. F. SMITH (Ind. Eng. Chem., 1927, **19**, 801—803).—The changes in free energy involved in the formation of methane, ethane, propane, or benzene, and methyl, ethyl, or propyl alcohols, from carbon monoxide and hydrogen, have been calculated as functions of the temperature, and the values of the equilibrium constants at 25°, 300°, and 400° have been deduced therefrom. A tendency is shown towards the formation of the higher rather than the lower members of the paraffin series, and of the aromatic rather than the straight-chain compounds. The lower alcohols cannot be formed from water-gas at atmospheric pressure and temperatures of 300° and above, whereas the production of considerable amounts of the higher alcohols is possible under these conditions. The tendency to form all these compounds falls off rather rapidly with rising temperature. A. B. MANNING.

Carbonisation of wood. I. Acids of crude pyroligneous acid. J. SEIB (Ber., 1927, **60**, 1390—1399).—Fractional distillation of the "residual acids" from pyroligneous acid is not a suitable process for the separation of the mixture into its components. Better results are obtained by use of the methyl or ethyl esters, but the isolation of homogeneous compounds depends mainly on individual processes. The following acids have been isolated: propionic, butyric, *iso*butyric, *iso*valeric, α-methylbutyric, *n*-hexoic, *iso*hexoic, heptoic, methacrylic, tiglic, and Δ^{α}-pentenoic. The presence of γ-butyrolactone, γ-valerolactone, and durene is established. The unsaturated acids all contain the double linking in the αβ-position, and where stereoisomerism is possible only the *trans*-form appears present. Angelic acid, obtained by Krämer and Grodzki, is a mixture of tiglic and Δ^{α}-pentenoic acids, whilst the *iso*crotonic acid of these authors is a mixture of solid crotonic acid, α-methylbutyric acid, and *iso*valeric acid. H. WREN.

Unsaturated hydrocarbons in oils obtained from petroleum. G. GANE and (Mrs.) M. ZILISTEANU-GHEORGHIU (Bul. Soc. Chim. România, 1926, **8**, 3—10).—Values of d^{15}, ignition temperatures, viscosities, congelation temperatures, and iodine values of a series of oils from the Steaua Româna and Vega refineries have been determined and the results tabulated. Two methods are described for determining amylene in presence of hexane, benzene, and mesitylene, and it is found that light petroleum, d 0·730, contains 12·5% of unsaturated hydrocarbons. H. BURTON.

Polymerisation and other chemical reactions in the sulphuric acid refining of cracked distillates. J. C. MORRELL (Ind. Eng. Chem., 1927, **19**, 794—801).—Determinations have been made of the losses due to polymerisation and dissolution which occur during the refining of cracked distillates from Californian and Mid-continent oils with different amounts of sulphuric acid. Using 1·55% by vol. of acid, the polymerisation losses are less than 2% and 1% (calculated on the gasoline fraction) respectively, with cracked distillates from the two types of crude oil. A negative polymerisation loss observed with cracked distillates from the Cushing field is attributed to the polymerisation of very low-boiling hydrocarbons which are lost during fractionation of the untreated distillate. The sum of the dissolution losses on acid treatment of the gasoline and gas-oil fractions of a Mid-continent cracked distillate agrees with the loss on treatment of the original distillate before fractionation, but the corresponding sum in the case of the Californian distillates is greater than the direct dissolution loss. Sulphuric acid treatment causes no appreciable change over the greater part of the distillation curves of Californian cracked gasolines, but produces a definite displacement (towards higher b.p.) of the curve for Mid-continent oils. In all cases there is a pronounced rise of the end b.p. of the treated gasoline, due, not only to polymerisation, but to the formation of sulphuric acid esters, which decompose on distillation, giving high-boiling, tarry residues. Sulphuric acid refining of cracked gasolines results mainly in a decrease in the percentage of unsaturated hydrocarbons, the content of aromatic, naphthenic, and paraffin hydrocarbons being only indirectly affected.
A. B. MANNING.

Specific and sensible heats of petroleum oils. O. G. WILSON, JUN. (Ind. Eng. Chem., 1927, **19**, 824—825).—Nomographic charts have been constructed to give the sp. ht. and the sensible heat above 0° F. of petroleum oils as functions of the temperature and the sp. gr. They are based on the formula of Fortsch and Whitman (B., 1926, 810). A. B. MANNING.

Refractometric examination of liquid fuels. K. R. DIETRICH (Chem.-Ztg., 1927, **51**, 509—510).—Results of the refractometric examination of a series of mixtures of petrol, benzine, and benzol with an immersion refractometer are described. Since petrol has the lowest refractive index, addition of either benzine or benzol or mixtures of the two can be easily detected and measured by a refractometric determination. Precautions to be observed in the determination are stated, and a simple apparatus for the circulation of warm water is described. E. H. SHARPLES.

Direct production of end-point petrol from cracking plants. K. NEUBRONNER (Chem.-Ztg., 1927, **51**, 508—509).—The conditions necessary for the production of cracked spirit free from resinifying and acid constituents and requiring no more refining are discussed.
E. H. SHARPLES.

Determination of hard asphaltum in cylinder oil. A. BOURGOM (Bull. Féd. Ind. Chim. Belg., 1927, **6**, 201—204).—The amount of asphaltum precipitated from mineral oils by benzine is greater the richer the benzine is in light constituents. The benzine can be used repeatedly provided the percentage of volatile constituents is not too high. When asphaltum is determined by Holde's method ("Kohlenwasserstoffe und Fette") it is thus essential to know the distillation limits of the benzine used. S. K. TWEEDY.

Apparatus for gas analysis. PERQUIN.—See I.

Gas analysis apparatus. FREDERICK.—See VI.

Penetrance of oils in wood. HOWALD.—See IX.

PATENTS.

Manufacture of granular carbon. STANDARD TELEPHONES AND CABLES, LTD. From WESTERN ELECTRIC CO., INC. (E.P. 272,279, 8.3.26).—A carbonaceous material is distilled at 800—1100° in an atmosphere of hydrogen at a rate such that low porosity is induced in the granular product, which is then heated at a higher temperature so that a roughened surface is imparted by the chemical action of an atmosphere of controlled oxidising power. The granular carbon is for use in microphones. R. A. A. TAYLOR.

Reactivating exhausted, pulverulent, decolorising charcoal. VEREIN F. CHEM. U. MET. PROD. (E.P. 264,799, 16.11.26. Conv., 22.1.26).—The charcoal is agglomerated in a press, the foreign substances (sugar etc.) acting as binding material, and is then ignited in a suitable furnace, such as a vertical shaft furnace, the ignited product being washed in water or dilute hydrochloric acid. W. G. CAREY.

Method and apparatus for cooling coke. KOPPERS COKE OVEN CO., LTD. From H. KOPPERS (E.P. 271,327, 29.10.26).— The coke is fed into a vertical shaft of such depth that the cooled coke prevents ingress of air or loss of gases. The coke is cooled by inert gases being blown transversely through the centre of the coke column, the partially heated gases being then withdrawn and blown through the top of the coke column, where they attain a temperature of 700—800° and pass to a waste-heat boiler. A. C. MONKHOUSE.

Drying coke and the like. H. FLEISSNER (U.S.P. 1,632,829, 21.6.27. Appl., 11.1.26. Conv., 14.8.24).—The coal is heated for 1 hr. in the presence of steam at a pressure of from 3 to 25 atm. corresponding to 130—235°. The pressure is then gradually reduced until the coal is dry. A. C. MONKHOUSE.

Manufacture of water-gas. HUMPHREYS & GLASGOW, LTD., and J. C. STELFOX (E.P. 272,026, 18.5.26).—Where the "back-run" method of operating a gasification plant is adopted (cf. E.P. 246,970; B., 1926, 477), in addition to a limited back-run with steam superheated in the recuperators, gas alone is circulated by fan or blower through the recuperators and the entire

fuel bed of the generator. Steam may be added at the inlet to the generator if desired. A. C. MONKHOUSE.

Distillation of solid fuels. C. STILL (E.P. 272,375, 20.10.26).—The yield of volatile products from a charge undergoing carbonisation in intermittently operated and laterally heated retorts is improved by the withdrawal by suction of the vapours. This is effected through suitable tubes or channels, the entrance to which is located below the surface of the coal charge, so that the vapours are drawn through the interior of the charge. R. A. A. TAYLOR.

Manufacture of liquid and other hydrocarbons and derivatives thereof from coal and like materials. I. G. FARBENIND. A.-G. (E.P. 247,582, 247,587, 250,948, 272,829, 272,834—5, [A, B, D, E], 11.2.26, [C, F], 14.4.26. Conv., [A], 14.2.25, [B], 16.2.25, [C], 15.4.25. Addn. [B—F] to E.P. 247,582).—(A) Solid carbonaceous materials are treated at an elevated temperature and under at least 50 atm. pressure with hydrogen or reducing gases containing hydrogen compounds. Nitrogen compounds, with or without other catalysts, are introduced. Ammonia, for example, acts catalytically, and as a reducing agent. The gases may be passed as a stream. By admitting steam, hydrogen may be generated in the reaction vessel from coal, hydrocarbons, or carbon monoxide, with the last of which the employment of the gases in a stream is rendered imperative. The conditions may be regulated to promote the formation of nitrogen compounds from free nitrogen. (B) Distilled or extracted derivatives of coal etc. or their fractions are used as raw material instead of solid carbonaceous materials. (C) Mineral oils or bitumens or fractions thereof are used as raw material; but if the reducing gas is ammonia, some other nitrogenous catalyst, *e.g.*, silicon nitride, is necessary. (D) When solid catalysts or porous materials are used, they are arranged in a separate contact vessel placed after the reaction vessel, so that only the vapours of the resulting products reach them. (E) The raw material consists of products or their fractions obtained by the hydrogenation of solid carbonaceous materials. (F) Conversion or hydrogenation products of mineral oils and bitumens are used. R. A. A. TAYLOR.

Manufacture of liquid or other hydrocarbons and derivatives thereof from coal and like materials. I. G. FARBENIND. A.-G. (E.P. 247,583, 272,830, 272,833, 11.2.26. Conv., [A], 14.2.25. Addn. [B, C] to E.P. 247,583).—(A) Solid carbonaceous materials are treated, at an elevated temperature and under at least 50 atm. pressure, with hydrogen or reducing gases containing hydrogen, in the presence of molybdenum or its compounds, mixed, if desired, with other catalysts or inert materials (*e.g.*, iron, calcium carbonate). (B) The molybdenum catalyst is assisted in its action by zinc oxide, magnesia, silica, coke, or activated carbon, the gas being passed in a stream. (C) Tarry products obtained by destructively hydrogenating solid carbonaceous materials are used as raw material, and the molybdenum catalyst is assisted by silicate of aluminium or magnesium or a mixture of these. R. A. A. TAYLOR.

Manufacture of hydrocarbons and derivatives thereof from coal and like solid materials. I. G. FARBENIND. A.-G. (E.P. 247,584—5, 272,831—2, 11.2.26. Conv., [A], 14.2.25, [B], 16.2.25. Addn. [B—D] to E.P. 247,584).—(A) Solid carbonaceous materials are treated at an elevated temperature and under at least 50 atm. pressure with hydrogen or reducing gases containing combined hydrogen, sulphur compounds (*e.g.*, the sulphides of the heavy metals, especially of the iron group) being admixed as catalysts. (B) Instead of solid carbonaceous material, distilled or extracted derivatives thereof are used as raw material. The hydrogenating gases may be used in the form of a stream. (C) In using a stream of gases the process can be rendered continuous. (D) The products of destructive hydrogenation of solid carbonaceous materials and products distilled or extracted therefrom, *e.g.*, coumarone resins and waxes from lignite, serve as raw material. R. A. A. TAYLOR.

Converting high-boiling hydrocarbons, which have been freed from the substances soluble in liquid sulphurous acid, into low-boiling hydrocarbons by means of aluminium chloride. ALLGEM. GES. FÜR CHEM. IND. M.B.H. (E.P. 271,042 and 272,433, 11.3.27. Conv., [A], 12.5.26; [B], 19.6.26).—(A) The products obtained by freeing high-boiling hydrocarbons from constituents soluble in liquefied sulphur dioxide or by splitting with aluminium chloride or double compounds of aluminium chloride (Edeleanu process) are (A) first of all split up by the action of small quantities of aluminium chloride, and the product so obtained is converted into low-boiling hydrocarbons by further treatment with aluminium chloride or worked up into marketable oils by the usual refining agents; or (B) subjected to renewed treatment with liquid sulphur dioxide, which process, after further treatment of the product with small quantities of aluminium chloride, is repeated until most of the Edeleanu refined product is converted into light hydrocarbons. R. A. A. TAYLOR.

Fractional distillation and condensation [of oil]. J. E. BELL, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,619,396, 1.3.27. Appl., 14.10.21).—Oil to be distilled cascades through a series of stills at successively higher temperatures. The vapours from each still preheat the oil flowing into that still, and the condensed vapours are separated. The uncondensed vapours from each preheater are utilised to heat the still at the next lower temperature. S. PEXTON.

Production of bitumen or oil emulsions. H. E. POTTS. From MINERAL A.-G. BRIG (E.P. 271,177, 24.2.26).—The emulsions are prepared from any bituminous substance, but reference is made to "Mexphalte" and "Spramex." The bitumen is mixed with sulphurised factice oil, fatty acids, or resins, such as are obtained by the action of sulphur chloride on train oil, fatty acids, or resins, or from linseed oil and sulphur heated at 150—180°. The mixture is emulsified with alkali in a high-speed colloid mill. In certain cases alkali is added so that only a portion of the oil added is saponified, the remainder in the presence of air and sunlight being converted into an asphalt. Lignite, brown coal, or alkali extracts of these may be also added. The emulsion is diluted with soap solution or other saponification product of a complex fatty amino-acid. A. C. MONKHOUSE.

Working-up acid resins, obtained from the refining of mineral oil derivatives, into natural bitumens. F. WILHELM (E.P. 254,674, 26.11.25).—The raw acid resins, produced in the refining of mineral oils by sulphuric acid washing, are heated in agitators, the sulphuric acid which separates being drawn off in regulated quantity so as to leave 1—3% in the resin. After further heating at 130°, a stream of hydrogen sulphide is passed in or sufficient sulphur is added to reduce the sulphuric acid to sulphur, which reacts with the resin forming a bituminous product, the hardness of which can be controlled by variation of the proportion of acid left in the resin prior to the reduction stage.
S. PEXTON.

Treatment of petroleum products. J. C. BLACK, W. D. RIAL, and R. T. HOWES, Assrs. to PAN AMERICAN PETROLEUM CO. (Re-issue 16,679, 26.10.26, of U.S.P. 1,592,329, 1.6.25).—See B., 1926, 863.

Separating water and other impurities from mineral and other oils or liquids. H. J. HOLFORD, Assr. to P. P. HARVEY (U.S.P. 1,635,845, 12.7.27. Appl., 9.9.25. Conv., 8.11.24).—See E.P. 238,750; B., 1925, 838.

Purification of liquid hydrocarbons. ALLGEM. GES. FÜR CHEM. IND. M.B.H. (E.P. 258,846, 11.8.26. Conv., 26.9.25).—See G.P. 432,580; B., 1927, 210.

Air-gas producing apparatus. L. BRÉGEAUT (E.P. 264,482, 7.1.27. Conv., 14.1.26).

[Valve for] gas-retort plant. R. DEMPSTER & SONS, LTD., and H. J. TOOGOOD (E.P. 273,063, 19.5.26).

Gas analysis apparatus (E.P. 257,609 and 272,092).—See I.

Distillation of oils (G.P. 439,044).—See III.

III.—TAR AND TAR PRODUCTS.

Solubility of naphthalene. G. WEISSENBERGER (Z. angew. Chem., 1927, **40**, 776).—Tables and curves for the solubilities of naphthalene expressed in g. of the solid per 100 g. of solution, at temperatures from — 14° to 50°, in various solvents are given. The best solvent is tetralin, 100 pts. of the solution containing 30 pts. of the solid at 13°, and 60 pts. at 60°; then, in order of solvent power come decalin, hexalin, and methylhexalin.
S. I. LEVY.

Distinguishing tests for carbolic acid, the cresols, and certain other phenols. A. H. WARE (Analyst, 1927, **52**, 335—337).—One drop of the phenol is dissolved in 10 c.c. of hydrochloric acid, 0·5 g. of a mixture of sodium nitrite (1 pt.), potassium or sodium nitrate (1 pt.), and dehydrated sodium sulphate (2 pts.) is added, and the whole stirred. After 2—5 min. the colour (*a*) is noted and then 1 c.c. of the acid mixture is poured into excess of 10% ammonia solution, and any colour change (*b*) again noted. Carbolic acid gives a rich crimson colour (*a*), changing on addition of 1—2 drops of 38% formaldehyde to purple, which on pouring into ammonia solution becomes deep blue. *o*-Cresol gives (*a*) a dichroic solution with green predominating, changing with formaldehyde to blue and with ammonia solution to olive-green. Both *m*- and *p*-cresol and "Cresol B.P." fail to give any distinctive results, and *p*-cresol inhibits the reaction with carbolic acid or *o*-cresol. β-Naphthol and α-naphthol give for (*a*) a crimson-purple and violet-purple, respectively, and for (*b*) the colour is destroyed. Thymol gives (*a*) a green and (*b*) a yellow colour. "Cresol B.P." gives similar results to *m*-cresol and "Creosote B.P." to guaiacol.
D. G. HEWER.

Detection of carbolic acid in commercial cresols. A. H. WARE (Pharm. J., 1927, **118**, 775—776).—The B.P. test for the detection of phenol in cresols is stated to be useless, and the following is advised: the cresol is shaken with 0·1*N*-potassium hydroxide, and, after separating, the upper layer is removed, shaken with ether, acidified after removal of the ether, and finally extracted with ether. The residue after evaporation of the second ether extract is acidified and stirred with a mixture of sodium nitrate and nitrite, the presence of phenol being shown by a crimson coloration (cf. preceding abstract).
B. FULLMAN.

Determination of phenol and cresol [in lysol]. K. K. JÄRVINEN (Z. anal. Chem., 1927, **71**, 108—117).—Determination of cresols gravimetrically by extraction with ether is unsatisfactory, because it is difficult to dry to constant weight. In the cold, cresol takes up, in general, two atoms of bromine, but the amount increases with the excess of bromine used. The following method is recommended for the determination of cresols in lysol. The fatty acids in a solution of 10 g. of 50% lysol in 100 c.c. of water and 30 c.c. of 2*N*-potassium hydroxide are precipitated with 30 c.c. of *N*-barium chloride solution, and the mixture is made up to 200 c.c. To 5 c.c. of the clear liquor are added 120 c.c. of water, 50 c.c. of 0·2*N*-bromide–bromate solution, and 25 c.c. of 2*N*-hydrochloric acid. After ½ hr., 10 c.c. of a 20% potassium iodide solution are added, and the iodine liberated is titrated with 0·2*N*-thiosulphate. The bromine number of the mixture of cresols in the lysol is determined by extracting an acidified fresh portion of 50 c.c. of the solution twice with 25 c.c. of a mixture of equal parts of ether and benzine of b.p. 50°, driving off the solvent on the water-bath, drying for ½ hr. at 100°, and weighing, then brominating one-tenth of the product exactly as described above. The error of the method is about 1%. The iodine method of Messinger and Vortmann also gives satisfactory results provided that the solution is kept cold and is first acidified and then neutralised with magnesia, so that the alkalinity is always the same.
R. CUTHILL.

PATENTS.

Refining tars. SIEMENS UND HALSKE, A.-G., Assees. of C. HARRIES (G.P. 439,005, 22.7.22).—The tars, or tar oils, are mixed with higher alcohols, *e.g.*, amyl alcohol, or their derivatives, and the mixture is extracted with alcohol. The refined oil and the extract are freed from alcohols by distillation *in vacuo*, or at the ordinary pressure, with or without steam.
A. B. MANNING.

Continuous distillation of tars and oils. R. BLÜMNER (G.P. 439,044, 4.2.25).—In the cracking of tars and oils by passage of the liquid through molten metal, the heated liquid is led from the autoclave to a well-lagged pressure container; it vaporises only on being

discharged from this container, and is then fractionally condensed. A. B. Manning.

Separation of tars, tar oils, etc. into fractions. J. A. Duijts (Dutch P. 15,083, 5.12.24).—The material is mixed with alkali and oils rich in phenols, which, *e.g.*, like low-temperature tar, are miscible with tar and tar oils. The mixture is then allowed to separate out, the various layers being drawn off and worked up independently; or the mixture may be separated by centrifuging. A. B. Manning.

Production of lower-boiling oils from low-temperature tars or tar oils. Comp. des Mines de Vicoigne, Noeux, et Drocourt (F.P. 614,229, 1.7.25).—The tars or tar oils, freed from phenols, are distilled in the presence of aluminium chloride. A. B. Manning.

Distillation of tar. J. S. Morgan and D. Rider, Assrs. to Thermal Industrial & Chemical (T.I.C.) Research Co., Ltd. (U.S.P. 1,635,896, 12.7.27. Appl., 1.6.22. Conv., 7.6.21).—See E.P. 184,624; B., 1922, 803 A.

IV.—DYESTUFFS AND INTERMEDIATES.

Phenol fusion. F. H. Rhodes, D. W. Jayne, jun., and F. H. Bivins (Ind. Eng. Chem., 1927, **19**, 804—807).—The effect of varying the temperature, amount of caustic soda, and time of heating, on the yield of phenol from the reaction between sodium benzenesulphonate and fused caustic soda, has been studied. The optimum conditions, giving a yield of 96% of the theoretical, are (*a*) a temperature of 350°, (*b*) an amount of soda 15% in excess of the theoretical, and (*c*) a 15-min. period of heating after mixing. Air must be excluded from the melt, and any local excess of sulphonate during mixing must be avoided. Among the oxidation products formed in the presence of air are the dihydroxydiphenyls and carbon dioxide, whilst, in the presence of excess of sulphonate, phenyl ether and thiophenol are produced in considerable quantities. A. B. Manning.

Patents.

Manufacture of azo dyes. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 248,766, 5.3.26. Conv., 5.3.25. Addn. to E.P. 231,529; B., 1925, 909).—Bordeaux dyes, yellower than those of the prior patent, are obtained in substance or on the fibre by coupling a 2:3-hydroxynaphthoic arylamide with a diazotised 2-amino-4-aroylaminodiaryl ether. Thus, 2-amino-4-benzamidodiphenyl ether is diazotised and coupled with 2:3-hydroxynaphthoic 5-chloro-*o*-tolylamide or α-naphthylamide. C. Hollins.

Manufacture of azo dyes. O. Y. Imray. From I. G. Farbenind. A.-G. (E.P. 272,580, 9.3.26).—*m*-Nitro-*p*-aminophenyl ethers (especially methyl and ethyl ethers) are diazotised and coupled with an acetoacetanilide containing an *o*-substituent or a *p*-alkoxyl group, giving yellowish-red pigments suitable for lakes. The second components used in the examples are the *o*-toluidide, *p*-anisidide, *m*-4-xylidide, and *2-chloro-4-anisidide* (m.p. 109—110°, made from *o*-chloro-*p*-anisidine), of acetoacetic acid. C. Hollins.

Manufacture of anthraquinone derivatives. I. G. Farbenind. A.-G., Assees. of J. D. Riedel A.-G. (E.P. 248,759, 3.3.26. Conv., 3.3.25).—Hydrogenation of anthraquinone or its derivatives in the liquid phase (preferably in a solvent or diluent) in presence of a catalyst (especially the mixed catalyst of G.P. 369,374; cf. B., 1923, 543 A) leads successively to the corresponding anthranols, tetrahydro- and octahydro-anthranols. The preparation of anthranol, m.p. 163—165°, *tetrahydroanthranol*, m.p. 108° (*benzoate*, m.p. 142°; *acetate*, m.p. 109°; *monobromo*-derivative, m.p. 123°), and *octahydroanthranol*, m.p. 124° (*acetate*, m.p. 52°; *benzoate*, m.p. 128°; *monobromo*-derivative, m.p. 123°), are described. Tetrahydroanthraquinone (Schroeter, A., 1925, i, 127) and *octahydroanthraquinone*, m.p. 180°, are obtained by oxidation of the respective anthranols. C. Hollins.

Manufacture of anthraquinone paste. W. M. Grosvenor and V. P. Gershon, Assrs. to W. M. Grosvenor (U.S.P. 1,631,589, 7.6.27. Appl., 10.10.21).—Crude anthraquinone is sublimed in steam (3 pts.), being rapidly condensed by treatment with atomised water (20 pts.). A stable homogeneous paste containing about 20% of pure anthraquinone is obtained. T. S. Wheeler.

Recovery of antimony in flavanthrone manufacture. E. C. R. Marks. From E. I. Du Pont de Nemours & Co. (E.P. 272,597, 15.3.26).—The filtrate from the flavanthrone is distilled at ordinary or reduced pressure, and to the distillate, consisting of nitrobenzene, chlorinated nitrobenzene, and antimony trichloride, enough dry chlorine is added below 50° to convert the trichloride into pentachloride. The mixture is then used for further batches of flavanthrone. The recovery of nitrobenzene is 80%, of antimony pentachloride 80—85%. C. Hollins.

Manufacture of amines of the cyclohexane series. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 261,747, 16.11.26. Conv., 17.11.25).—Secondary *cyclo*hexylarylamines, obtained from *cyclo*hexyl halides and arylamines, are alkylated, *e.g.*, with methyl or ethyl sulphate, chloride, or *p*-toluenesulphonate. cyclo*Hexylmethylaniline*, b.p. 148—150°/12 mm., and cyclo*hexylethylaniline*, b.p. 150—155°/12 mm., are described. The products are intermediates for basic and sulphide dyes. C. Hollins.

Pyrazolone dyes from amino-derivatives of 4-hydroxy-3-carboxydiphenyl sulphide. British Dyestuffs Corp., Ltd., and M. Mendoza (E.P. 272,024, 12.5.26).—The utilisation of the screening effect of a sulphide bridge (cf. E.P. 260,058; B., 1927, 8) between the chromophoric and the chelate parts of the molecule is extended to the preparation of pyrazolones from hydrazino-4-hydroxy-3-carboxydiphenyl sulphides, which, when coupled with diazo compounds, give dyes practically unchanged in shade by chroming. Diazotised 2′-amino-4-hydroxy-3-carboxy-4′-sulphodiphenyl sulphide, for example, is reduced to the hydrazine and condensed with ethyl acetoacetate to give a methylpyrazolone, which is coupled with diazotised sulphanilic acid (greenish-yellow after-chromed on wool, or chrome-printed on cotton), 2:5-dichloroaniline-4-sulphonic acid

(redder shades), naphthionic acid (yellowish-orange), aminoazobenzene (orange), benzene-4-azo-1-naphthylamine-6(or 7)-sulphonic acid (yellowish-red after-chromed on wool, red-brown chrome-printed on cotton), or with tetrazotised benzidine (orange after-chromed on wool, light brown chrome-printed on cotton). The corresponding pyrazolonecarboxylic acid gives redder shades.
C. HOLLINS.

Preparation of triarylmethane dyes. BRITISH DYESTUFFS CORP., LTD., E. H. RODD, and F. W. LINCH (E.P. 272,321, 17.4.26).—Tetra-alkyldiaminodiaryl ketones combine with sodium (2 atoms), in presence or absence of a solvent, to form derivatives, which, by reaction with aryl halides, yield the carbinol bases of triarylmethane dyes: $R_2CO \rightarrow R_2CNa \cdot ONa \rightarrow R_2R'C \cdot ONa$. The reactions may be performed in one operation, the halide being added after the sodium has dissolved. 4 : 4'-Tetramethyldiaminotriphenylcarbinol and the tetraethyl homologue are obtained from tetramethyl- and tetraethyldiaminobenzophenones, respectively, with chlorobenzene; with β-chloronaphthalene, *carbinols* melting, respectively, at 181° and 175—177° result. From tetramethyldiaminobenzophenone with *o*-chlorotoluene a *carbinol*, m.p. 163°, is prepared; with 4-chloro-*m*-xylene, a *carbinol*, m.p. 145°. The salts of these carbinols are green or blue-green dyes. C. HOLLINS.

Manufacture of acid dyes. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 252,390, 21.5.26. Conv., 22.5.25).—A 1 : 3-dihalogeno-4 : 6-dinitrobenzene is condensed with 2 mols. of a *p*-aminodiphenylaminesulphonic acid to give yellow to brown dyes for animal fibres; other substituents may be present. Thus 1 : 3-dichloro-4 : 6-dinitrobenzene with 4'-nitro-4-aminodiphenylamine-2'-sulphonic acid gives a yellow; with 4-aminodiphenylamine-2-sulphonic acid, a brownish-yellow; with 4-amino-4'-methyldiphenylamine-2-sulphonic acid, an orange-yellow. 2 : 6-Dichloro-3 : 5-dinitrotoluene with 4-aminodiphenylamine-2-sulphonic acid gives a brown. The dyeings on wool are fast to light and fulling.
C. HOLLINS.

Sulphur-black dye. O. ZELLER and C. WALDMAN, Assrs. to NATIONAL ANILINE & CHEMICAL CO., INC. (U.S.P. 1,630,818, 31.5.27. Appl., 12.5.22).—Sulphur-black dyes, obtained, *e.g.*, by the action of sodium polysulphide on sodium dinitrophenoxide, are mixed in the crude moist condition with an excess of sodium sulphide, and dried under reduced pressure to give a stable product.
T. S. WHEELER.

Sulphonic acids (E.P. 253,118).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Technical use of chlorine for the decomposition of raw vegetable fibres. P. WAENTIG, also H. WENZL (Papierfabr., 1927, 25, 340—341, 341—342; cf. B., 1927, 69, 292).—(A) In reply to Wenzl the author confirms his earlier statements regarding (*a*) the quantity of heat developed in the chlorination of pine-wood meal, (*b*) the resolution of pine wood by chlorine under pressure into bleachable cellulose of high α-cellulose content, and (*c*) the ratio of hydrochloric acid to combined chlorine in the chlorination of pine wood with chlorine gas and chlorine water. The lower yields of pulp recorded by Wenzl in the chlorination of straw with chlorine water as compared with chlorine gas are attributed to losses in washing and not to any specific differences between the two methods. (B) Waentig's conclusions are, in the author's opinion, of more theoretical than practical value. Experiments on a semi-commercial scale have proved that, in practice, lower yields of straw pulp are obtained with chlorine water than with chlorine gas.
D. J. NORMAN.

Pine wood without lignin reactions. W. FUCHS (Ber., 1927, 60, 1327—1330; cf. B., 1927, 405).—The preparation of pine wood without lignin reactions is easily effected without considerable loss in weight by oxidation with perbenzoic acid in ordinary daylight. Oxidised wood yields more dextrose than ordinary wood and contains less lignin. The increase in the former appears to depend on increase in the cellulose content. The lignin of oxidised wood does not differ greatly in elementary composition from hydrochloric acid lignin, but contains less carbon and considerably less methoxyl than the latter. With molten potassium hydroxide it affords one and a half times as much protocatechuic acid as ordinary lignin; it yields very little hydroxymethylfurfuraldehyde when treated with superheated steam. H. WREN.

Some factors in the copper number of cellulose. H. LE B. GRAY and C. J. STAUD (Ind. Eng. Chem., 1927, 19, 854—855).—Results obtained in the copper number determination are influenced by the rate of stirring, *e.g.*, a sample of degraded cellulose gave with gentle stirring 1·76, and without stirring 1·92. The difference is not due to aerial oxidation, since similar figures are obtained when determinations are carried out in an atmosphere of nitrogen. The copper number is also influenced by the degree of subdivision of the pulp. A sample of sulphite pulp had a copper number of 1·64 when cut into 6 mm. squares, and 1·40 when rasped. This result may be due to preferential absorption of alkali from the Fehling's solution, followed by the destruction of the reducing groups of the cellulose before contact with cupric ions is obtained, such preferential absorption being favoured by increase in the surface exposed. This view is supported by the observation that if sulphite pulp is heated with the alkaline tartrate solution for 45 min. at 100° before the copper sulphate is added, and from this point the copper number determination is carried out as usual, the result is approximately 50% of the normal figure, showing that the alkaline tartrate solution has destroyed the reducing groups to a considerable extent. W. J. POWELL.

Hydrolysis number determination for wood cellulose. L. F. HAWLEY and L. C. FLECK (Ind. Eng. Chem., 1927, 19, 850—852).—In the determination of hydrolysis numbers, 1 g. of the α-cellulose (Cross and Bevan method) from the pulp is heated with 100 c.c. of 15% sulphuric acid for 3 hrs. on a boiling-water bath, the loss in weight (%) being the hydrolysis number. The yield of pulp in a given sample of wood is invariably lower than the α-cellulose content of the latter owing to the hydrolysis suffered by the cellulose during the pulping process. From determinations of the α-cellulose content

and of the hydrolysis number of the α-cellulose, carried out on the original wood and on the pulp, the yield of the latter may be calculated, and the figures obtained agree well with experimental results. W. J. POWELL.

Effect of certain organic bases in plasticised nitrocellulose films. L. L. STEELE (Ind. Eng. Chem., 1927, **19**, 807—808).—The effect of the addition of 1% of various organic nitrogen compounds to a lacquer on the life of the film when exposed to the weather has been studied. The stronger bases, *e.g.*, *iso*allylamine, benzylamine, piperidine, were detrimental to the film, whilst others, *e.g.*, carbamide, appeared to have no action. The most effective compound in prolonging the life of the nitrocellulose film was diphenylamine (from 80 days for the standard film, to over 170 days), although the brown coloration produced in such a film on exposure to sunlight would be a drawback to its use as a stabiliser in commercial lacquers. A. B. MANNING.

***Alpina nutans* for paper-making.**—(Bull. Imp. Inst., 1927, **25**, 122—126).—The dried, fibrous stems of *A. nutans*, which contain a large amount of pithy matter, have the following composition: moisture 9·0%, ash 6·25%, cellulose 49·9%, cellulose in moisture-free material 54·8%, the average dimensions of the ultimate fibres being: length 2·62 mm., diameter 0·0162 mm. The character of the pulp produced by digestion with caustic soda under different conditions is described, and it is concluded that the stems can be converted into paper of very satisfactory quality, the yield of pulp being good and the consumption of caustic soda not excessive. E. H. SHARPLES.

Uses of artificial silk in the textile industries. P. E. KING (J. Soc. Dyers and Col., 1927, **43**, 219—225).

Impregnation of wood and removal of ash. BECHHOLD and HEYMANN.—See IX.

Formaldehyde in wood smoke. CALLOW.—See XIX.

PATENTS.

Fulling of animal fibres. I. G. FARBENIND. A.-G. (E.P. 270,333—4, 18.1.26. Conv., 21.1.25).—(A) Butylated naphthalenesulphonic acid or a salt thereof is used as a wetting agent in the acid fulling of animal fibres. (B) The wetting agent specified in (A) is replaced by an aliphatic sulphonic acid, *e.g.*, sulphonated brown coal tar oils or a salt thereof. D. J. NORMAN.

Carroting fur. Preparation of fur for shrinking and felting. J. H. MARTIN (U.S.P. 1,630,633—4, 31.5.27. Appl., [A], 17.12.24; [B], 3.3.25).—The fur is prepared for felting by treatment with (A) a solution of a peracid salt, *e.g.*, ammonium persulphate, or (B) sodium orthoperborate. T. S. WHEELER.

Preparing hair for felting. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 250,575, 1.4.26. Conv., 9.4.25).—Metal salts such as bismuth, zinc, or aluminium nitrate can satisfactorily replace mercury salts as carroting agents in the manufacture of felt if they are used in conjunction with wetting agents such as Leonil, Nekal, or Neomerpin (sulphonic acids of alkyl-, aryl-, or aralkyl-naphthalene derivatives of high mol. wt.). D. J. NORMAN.

Weighting of fibres [silk]. O. BERG and M. IMHOFF (U.S.P. 1,634,012, 28.6.27. Appl., 25.2.26).—Silk fabrics are treated with a weak solution of acetic acid, then with a solution of lead acetate, and finally with a solution of sodium phosphate. W. G. CAREY.

Manufacture of cuprammonium silk threads. (Miss) W. SCHULZ (E.P. 249,845, 13.3.26. Conv., 26.3.25).—Cuprammonium silk filaments of high strength (20—21 kg./mm.^{2}) are obtained by spinning cuprammonium hydroxide solutions of cellulose containing not more than 3—4% (preferably 1—2%) of ammonia into a 1—2% aqueous solution of ammonia at 60—95°, and arranging that the length of travel of the thread in the coagulating bath shall exceed 1·5 m., *e.g.*, about 2·5 m. D. J. NORMAN.

Treatment of cellulose ester materials. R. CLAVEL (E.P. 252,661, 4.11.25. Conv., 30.5.25).—Material suitable particularly for use as a leather substitute is made by impregnating cellulose ester threads in hank or fabric form with solutions of benzenoid phenolic compounds, *e.g.*, tannins, and treating the impregnated material, preferably after washing, with salts of suitable metals, *e.g.*, bismuth, zinc, silver, or iron. The goods are then coloured, dried, calendered and treated with suitable finishing agents. D. J. NORMAN.

Production of material resembling celluloid. I. G. FARBENIND. A.-G. (F.P. 438,849, 26.10.26).—Nitrocellulose is kneaded with a phenoxyacetarylide alkylated at the nitrogen atom, in the presence of alcohol until gelatinisation is complete, the proportion of the arylide being insufficient for the production of a soft, elastic product; *e.g.*, 35 pts. of cresoxyethylacetanilide are used per 100 pts. of nitrocellulose. L. A. COLES.

Production of insulating paper containing phenol resins. FELTEN & GUILLEAUME CARLSWERK A.-G. (E.P. 270,243, 1.11.26. Conv., 29.4.26).—The water of the fibrous pulp is replaced by a solvent for phenol resins (*e.g.*, alcohol), a solution of the phenol resin added, and the well-mixed mass treated with water to precipitate the resin in and on the fibres. The material is then heated to convert the resin into the *C*-state or first worked up into paper or pressed articles before such conversion. B. P. RIDGE.

Improving the colour of pulp. F. G. RAWLING (U.S.P. 1,630,635, 31.5.27. Appl., 1.9.26).—The black colour of paper pulp containing iron compounds of tannic acid is lightened to brown by the addition of lime. T. S. WHEELER.

Manufacture of threads, filaments, strips, or films from cellulose esters. L. CLÉMENT and C. RIVIÈRE, Assrs. to COURTAULDS, LTD. (U.S.P. 1,634,980, 5.7.27. Appl., 17.11.24. Conv., 5.1.24).—See E.P. 224,404; B., 1925, 38.

Combining cellulose and rubber. S. A. OGDEN (E.P. 273,169 11.12.26).—See U.S.P. 1,617,495; B., 1927, 248.

Apparatus for drying woven and other fabrics. F. BROUGHTON and A. B. HENSHILWOOD (E.P. 270,430, 13.2.26).

Artificial leather (G.P. 437,078).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Dye solution. K. LÜTTIN, Assr. to J. R. GEIGY A.-G. (U.S.P. 1,635,628, 12.7.27. Appl., 8.6.23. Conv., 29.7.21).—See E.P. 183,813; B., 1923, 972 A.

Apparatus for the continuous [fluid] treatment of textile fibres in skein form. J. BRANDWOOD (E.P. 258,544, 17.3.26. Conv., 21.9.25).

Emulsions (E.P. 247,588).—See XX.

Sulphonic acids (E.P. 253,118).—See XX.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Oxidation of ammonia to nitrate at alkaline surfaces. K. A. HOFMANN [with K. LESCHEWSKI, W. LEMME, H. GALOTTI, K. MAYEN, and W. GUNDELACH] (Ber., 1927, **60**, 1190—1201; cf. A., 1926, 370).—Ammonia is quantitatively converted into a mixture of nitrite and nitrate in the presence of a moderate excess of air at a soda–lime surface below 310°. The rate of reaction is sufficiently great in the presence of small amounts of activators, such as nickel, copper, iron, manganese, cobalt, silver, or lead as metal or oxides. The direction of the change is controlled by the alkaline surface. The addenda do not function as oxygen carriers, and if present in more than small amount are disadvantageous, particularly if they diminish the alkalinity of the surface. The first isolable product of the change is nitrite, the precursor of which is regarded as imide. In favour of this view it is found that if a flame of hydrogen and ammonia burning in air is allowed to impinge on ice, nitrite is freely produced in the presence of alkali, but not of acid. The strongest evidence of the decomposition of ammonia, according to the schemes: $2HNH_2 + O_2 = 2NH + 2H_2O$ and $NH + O_2 + NaOH = NaNO_2 + H_2O$ is found in the observation that up to 3% of free hydrogen is present in the residual gases when a mixture of 1 vol. of ammonia and 1—10 vols. of air at temperatures below 320° is used, the imide thus decomposing in absence of sufficient oxygen into nitrogen and hydrogen. The oxidation of ammonia dissolved in water to nitrite at the atmospheric temperature is greatly activated by copper, particularly in the presence of alkali, but the other catalysts used above, except silver and its oxide, are ineffective. The oxidation of sodium oxide, sodamide, and hydroxylamine by molecular oxygen is much more vigorous, and does not require any activator.

H. WREN.

Colorimetric determination of perchlorate in saltpetre. O. S. FEDOROVA (J. Russ. Phys. Chem. Soc., 1927, **59**, 265—282).—The usual methods of determination are considered unsatisfactory. Monnier's colorimetric method (B., 1917, 49) was applied to the determination of perchlorate in the presence of nitrate; it was found that solutions of potassium nitrate (above 10%) and of sodium nitrate (above 5%) themselves form precipitates with methylene-blue. This method, as modified by Hofmann (B., 1926, 292), gave results accurate to within 0·02—0·03%, but is inconvenient as it depends upon temperature, length of time of reaction of perchlorate and methylene-blue, and the personal factor. Hahn's method (B., 1926, 404) is not only sufficiently accurate, but also rapid and simple.

J. KAYE.

Determination of sodium nitrite. F. A. HÖEG (Z. anal. Chem., 1927, **71**, 102—107).—Klemenc's method of determining nitrite (B., 1922, 963) has been modified as follows. The pressure within a flask containing 150 c.c. of a 20% solution of sulphuric acid and 50 c.c. of 0·1*N*-potassium permanganate is reduced to 10 cm., and 25 c.c. of a solution of 6·9 g. of commercial sodium nitrite in 1 litre of water are introduced through a tap-funnel. The mixture is heated at 40° for 5 min., with constant shaking, and then the stopper is removed and the excess of permanganate titrated with 0·04*N*-oxalic acid. This method gives more accurate results than the usual permanganate method, and the end-point is sharper. R. CUTHILL.

Dehydration of sodium sulphate decahydrate at the ordinary temperature. J. OBERMILLER (Z. angew. Chem., 1927, **40**, 755).—It is pointed out that anhydrous sodium sulphate is in equilibrium with air containing 63% of its saturation limit of water vapour at 24—25°, and that, therefore, the decahydrate of sodium sulphate will become completely anhydrous in air containing less moisture than this (cf. Rakusin and Brodski, B., 1926, 1011). A. R. POWELL.

Production of fluorite in the electric furnace. D. C. STOCKBARGER (J. Opt. Soc. Amer., 1927, **14**, 448).—Pure calcium fluoride is fused in an atmosphere of nitrogen and allowed to cool slowly. The solidified mass resembles a single large crystal with numerous fractures along its cleavage planes. W. E. DOWNEY.

Thiocarbamide as an impurity in thiocyanates. T. S. PRICE and J. W. GLASSETT (Phot. J., 1927, **67**, 329—330).—A specimen of potassium thiocyanate contained 0·2% of thiocarbamide, and a specimen of calcium thiocyanate contained the same impurity. The source of the impurity was probably ammonium thiocyanate in each case, thiocarbamide being formed when solutions of the latter are evaporated at too high a temperature. Silver sulphide being somewhat soluble in the ammonium salt, tests for thiocarbamide in this compound may fail. B. W. ANDERSON.

Detection of chlorides in mercuric oxide. G. J. W. FERREY (Pharm. J., 1927, **118**, 767—769).—The detection of less than 0·7% of chlorides (calculated as hydrogen chloride) in mercuric oxide by the addition of silver nitrate to the nitric acid solution is invalidated by the solubility of silver chloride in mercuric nitrate. Less than 0·02% of chlorides may, however, be detected by dissolving the mercuric oxide in dilute sulphuric acid (adding a drop or two of nitric acid), removing the mercury by addition of zinc turnings, and adding to the filtered liquid nitric acid and silver nitrate.

B. FULLMAN.

Modification of the Haldane general air analysis apparatus. R. C. FREDERICK (Analyst, 1927, **52**, 340).—A piece of capillary glass tubing bent at right angles with one arm about 1·5 cm. long and the other 5 cm. is attached by the shorter arm to the bottom of the

measuring burette of the Haldane apparatus, and by the longer arm to the rubber tube and mercury reservoir. By this means mercury cannot be carried over nor reagents drawn into the burette. D. G. Hewer.

Hempel gas analysis apparatus without absorption bulbs: its use in the examination of commercial oxygen. R. C. Frederick (Analyst, 1927, 52, 400—401).—The measuring burette of the Hempel gas apparatus has a simple stopcock at the top and a 3-way cock at the bottom, connected to a levelling burette, and, by an extra tube, to a small separating funnel which can be raised to the level of the upper stopcock. Details of operation vary with the nature of the sample, and in some cases it is possible to place the complete absorbing reagent in the funnel and collect the sample over water, but in the case of commercial oxygen the alkaline pyrogallol is formed *in situ*, thus preventing loss of absorptive power due to contact with atmospheric oxygen. D. G. Hewer.

Sodium selenite as a poison. Riechen.—See XIX.

Determination of mercury. Dunnicliff and Lal.—See XX.

Patents.

Still or column for the distillation of ammonia. W. T. Towler and R. Marsh (E.P. 272,641, 29.3.26).—The still consists of a hollow, cylindrical vessel, the upper and lower compartments of which contain a large number of small packing elements (Lessing packing), and an intermediate compartment milk of lime. Steam flows upwards through inlets at the bottom of the lower compartment and escapes with the ammonia vapour from an outlet on the top of the upper compartment, the ammonia liquor entering the upper compartment through a laterally disposed inlet and flowing downwards. In a modified form of the still the lower compartment is a separate vessel. W. G. Carey.

Production of ammonium sulphate. J. Meyrueis (F.P. 608,601, 31.3.25).—Ammonia gas or solution is added to aluminium sulphate solution, and, after removal of the precipitated aluminium hydroxide, the liquor is evaporated to dryness. L. A. Coles.

Apparatus for the production of ammonium sulphate from liquid ammonia. M. Mallet (F.P. 608,659, 8.4.25).—Liquid ammonia is conveyed through lead tubes from a closed chamber into an absorption vessel provided with a check valve, containing sulphuric acid. The upper side of the part of the tubes dipping into the acid is surrounded by a lead cylinder, and the lower side is pierced with fine holes for the passage of the ammonia. Means are provided for admitting air when the acid is neutralised. L. A. Coles.

Production of sodium sulphide. I. G. Farbenind. A.-G. (E.P. 264,464, 6.12.26. Conv., 18.1.26).—The reduction of sodium sulphate by carbon is effected in a furnace with a flat rotatable hearth rendered air-tight by providing it with a U-shaped collar filled with sand, into which dips a sheet-metal partition reaching from, and fastened to, the roof. The furnace charge is stirred by water-cooled ploughs fixed to a reciprocating arm. W. G. Carey.

Production of double salts of aluminium or other metals. G. Julien (F.P. 609,361, 14.1.26).—Salts suitable for rendering cement impervious to water are prepared by dissolving aluminium, zinc, iron, or magnesium, or their oxides, or mixtures of the metals or oxides, in a mixture or phosphoric and sulphuric acids, or in each acid separately, and subsequently mixing the solutions, and evaporating to dryness the solutions obtained. The aluminium salt has the formula $2AlPO_4,3Al_2(SO_4)_3,xH_2O$. L. A. Coles.

Production of alumina. A. L. Mond. From I. G. Farbenind. A.-G. (E.P. 272,748, 20.10.26).—Bauxite or clay is dissolved in sulphuric acid and the insoluble silicic acid separated. The solution is evaporated and the salts are heated to such a temperature that they decompose into oxides and acid, and the oxides of aluminium and iron thus freed from silicic acid are heated with a reducing agent in an electric furnace until the mixture is decomposed into alumina and metallic iron. W. G. Carey.

Production of cobaltous acetate. I. G. Farbenind. A.-G., Assees. of Chem. Fabr. Griesheim-Elektron (E.P. 262,075, 2.11.26. Conv., 27.12.25).—A mixture of cobalt hydroxide and acetic acid, the latter slightly in excess of the amount required theoretically to form cobalt acetate, is heated in an autoclave at 120° with a reducing agent, finely-divided cobalt being the most suitable. W. G. Carey.

Production of metal compounds. A. F. Meyerhofer (E.P. 272,733, 7.6.26. Addn. to E.P. 253,150; B., 1926, 1013).—Hydrofluosilicic acid, produced by the interaction of a sparingly soluble fluoride, silicon fluoride, and acid, is not used directly to reproduce barium silicofluoride but to produce some other silicofluoride as an intermediate step. C. A. King.

Production of phosphorus pentoxide or phosphoric acid. I. G. Farbenind. A.-G., Assees. of Chem. Fabr. Griesheim-Elektron (E.P. 260,225, 23.9.26. Conv., 22.10.25).—Molten yellow phosphorus is burnt by means of a nozzle or jet burner of the liquid fuel burner type, the phosphorus being supplied either from a vessel at a higher level than the burner, or by a pumping device. W. G. Carey.

Manufacture of phosphorus and phosphorus oxides. I. G. Farbenind. A.-G. (E.P. 264,520, 14.1.27. Conv., 15.1.26).—Raw phosphate, coal, and sand, or sand and alumina or fine clay are heated in a shaft furnace in such proportions that merely sintering and not fusing occurs at the reaction temperature of 1100—1400°, and the silicate residue is continuously or intermittently removed by a rotary or travelling grate. W. G. Carey.

Working with carbon monoxide under pressure. I. G. Farbenind. A.-G. (E.P. 247,217, 6.2.26. Conv., 7.2.25).—In the manufacture of methyl alcohol and allied products the carbon monoxide used is dried to prevent any reaction with the iron portions of the apparatus below 150°. Above 150° the apparatus is protected with linings of special alloys (cf. E.P. 231,285; B., 1925, 425). A. C. Monkhouse.

Liquefaction of air and separation into oxygen

and nitrogen. E. G. LUENING (U.S.P. 1,634,076, 28.6.27. Appl., 3.11.24).—A liquefying coil for the air is surmounted by a column of very much greater height than diameter which receives liquid from the coil through a connexion at the upper end of the column. Inside the column is a device for preventing convection currents in the liquid and for retarding the upward movement of bubbles from the surface of the coil through the liquid, and at the lower and upper ends of the column are means for indicating the height of the liquid column. W. G. CAREY.

Production of hydrocyanic acid. G. BREDIG and E. ELÖD (U.S.P. 1,634,735, 5.7.27. Appl., 28.1.24. Conv., 7.12.22).—See E.P. 229,774 and 229,973; B., 1925, 315.

Manufacture of cyanides. O. STALHANE (E.P. 272,996, 23.3.26).—See U.S.P. 1,610,897; B., 1927, 166.

Electrolysis of chlorides (E.P. 264,865 and 267,912). **Oxidation of nitrogen etc.** (U.S.P. 1,634,311).—See XI.

Hexamethylenetetramine and ammonium chloride (U.S.P. 1,630,782).—See XX.

Silver from thiosulphate solutions (Austr.P. 105,084).—See XXI.

VIII.—GLASS; CERAMICS.

Causes of plasticity of clay. H. SALMANG (Z. anorg. Chem., 1927, **162**, 115—126).—From a review of the various theories advanced from time to time, and the available data, it seems that the purely physical factors on which the plasticity of clay depends are the fineness, softness, and laminated structure of the particles, and the roughness of their surface. The other factor involved seems to be the formation of a viscous layer between the particles by a surface reaction between the liquid and solid phases. Thus, admixture of kaolin with hydrocarbons does not give a plastic mass, because no such reaction can occur. The rigidity of air-dry clay seems to be due to the colloids in the surface layers. In support of this view it is found that a sample of dry clay which has been moistened and then left in air till it has attained constant weight contains more moisture than a specimen which has been treated in the same way without being moistened. The superior rigidity in the air-dry state of poured ceramic masses compared with that of moulded masses appears to be due to a denser and more regular arrangement of the particles, and to the absence of air in the mass. R. CUTHILL.

Determination of titanic acid in refractory earths. L. LEMAL (Bull. Soc. chim. Belg., 1927, **36**, 395—396).—The sample is repeatedly extracted with hydrofluoric and nitric acids. The residue is ignited with a little ammonium carbonate, then fused with potassium hydrogen sulphate, and finally extracted with water and sulphuric acid. Ferric oxide and titanic acid are determined together by titrating the solution with potassium permanganate after reduction with zinc and acid. The ferric oxide alone is determined by titrating with potassium dichromate after reduction with stannous chloride. S. K. TWEEDY.

Determination of alkalis in clays etc. CIOCHINA.—See X.

PATENTS.

Preventing coloration of lime-soda glass. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY, & CIREY (E.P. 264,490, 10.1.27, Conv., 12.1.26).—In glasses such as those of the alkali-lime type, designed for use under exposure to sunlight or similar radiation, is incorporated up to 3% of lead oxide or 2% of titanium oxide, whereby the colour changes, produced by the action of short-wave radiation on iron, manganese, or selenium compounds present, are prevented. A. COUSEN.

Ceramic insulating material. Ceramic material. J. A. JEFFERY and E. T. MONTGOMERY, Assrs. to CHAMPION PORCELAIN CO. (U.S.P. 1,631,729 and 1,631,730, 7.6.27. Appl., [A], 24.2.19; [B], 24.5.19).—(A) A mixture of flint (10—15%), felspar (10—15%), talc (7—10%), calcium carbonate (5—10%), and kaolin, with or without addition of ball-clay, is formed, glazed, and fired to give porcelain of good insulating properties even at high temperatures. (B) A mixture of ball-clay, felspar, and kaolin fired with a pre-calcined mixture of kaolin, alumina, and talc yields a similar product. T. S. WHEELER.

[Manufacture of] porcelain. F. H. RIDDLE, Assr. to CHAMPION PORCELAIN CO. (U.S.P. 1,631,695, 7.6.27. Appl., 1.6.20. Cf. E.P. 259,757; B., 1926, 1014).—A mixture of kaolin and alumina is calcined to form synthetic sillimanite, which is incorporated with the product formed by calcining magnesium carbonate, kaolin, and flint, and fired with addition of ball-clay and kaolin to give a porcelain of good insulating properties even at high temperatures. T. S. WHEELER.

Manufacture of ceramic ware or materials. QUARTZ & SILICE (E.P. 263,765, 22.11.26. Conv., 24.12.25).—Amorphous silica, particularly previously fused silica, *e.g.*, silica waste from silica glass manufacture, is ground and used in place of crystalline silica in the manufacture of ceramic ware baked at a high temperature, such as electro-technical porcelain and faience. B. W. CLARKE.

IX.—BUILDING MATERIALS.

Colloidal theory of cements. T. MAEDA (J. Physical Chem., 1927, **31**, 933—936).—Previous work on the hardening and setting of magnesium oxychloride cement (cf. B., 1926, 587; 1927, 366) leads to the conclusion that both the crystal and colloid theories are necessary for a true explanation of the results. A new theory on these lines is advanced. L. S. THEOBALD.

Mineralogical content of Maine sands in relation to mortar strength. H. W. LEAVITT and J. W. GOWEN (Proc. Nat. Acad. Sci., 1927, **13**, 351—354; cf. B., 1927, 483).—An investigation of the dependence of the tensile strength of mortar briquettes composed of 1 pt. of Portland cement and 3 pts. of sand on the mineralogical character of the Maine sand used shows that increase in the content of granitic material (quartz, felspar, mica, etc.) results in a lowering of the mortar strength. Non-granitic sands usually contain the most iron, and the

presence of iron in such sands partly explains their greater strength in mortars. Iron content and granitic content, however, play a part independent of each other in determining the strength of a mortar resulting from a given sand. J. S. Carter.

Penetrance of oily fluids in wood. Neglected factors influencing penetration and absorption of creosotes, petroleum oils, and creosote-petroleum mixtures. A. M. Howald (Chem. and Met. Eng., 1927, 34, 353—355).—The penetrance of creosotes, petroleum oils, and creosote-petroleum mixtures in wood is not always determined by viscosity (other conditions being constant), since two oils of approximately the same absolute viscosity gave, when tested under the same conditions using matched samples of Douglas fir, absorption figures of 1·4 and 12·94 lb./cub. ft. respectively. D. J. Norman.

Impregnation of wood and removal of its "ash" by electrical means. H. Bechhold and E. Heymann (Z. Elektrochem., 1927, 33, 161—170).—Experiments have been made with logs of various woods about 20 cm. long and 7—10 cm. in diameter. For studying the removal of "ash" a potential difference was applied across carbon electrodes connected to the ends of the log by way of pads of wadding or felt kept moist by dipping into vessels of water. A notable transport of water from anode to cathode through the wood occurs and soluble salts are thus carried away, but acid substances produced by electrolytic action at the anode enter the wood in their place. By maintaining a continuous flow of distilled water over the electrode pads, however, efficient removal of the ash is possible, the ash content of a pine log falling in 15 days from 0·31 to about 0·02% using 0·4 kw.-hr. at 120 volts. With given applied voltage the current decreases during the process owing to removal of electrolytes, but the rate of transport of water—an electro-osmotic process—increases with decreasing conductivity. It is remarkable that in some cases the ash is removed more completely from the heart than from the sap wood. Removal of ash reduces the tendency of the wood to take up water, as measured by swelling when kept over 20% sulphuric acid solution, and, in general, reduces the intensity of growth of fungi on the wood. For studying the impregnation of wood the same arrangement was employed except that the anode pad dipped into a solution of the substance to be introduced into the wood. During impregnation with a metallic salt the rate of water transport through the wood decreases to a small constant value or even to zero, while the current rises, reaching a high value when metal begins to be deposited on the cathode. With mercuric chloride solutions the time required for mercury to be deposited on the cathode is much greater than that calculated from the porosity of the wood and the rate of water transport, suggesting strong absorption of this salt by the fibres, an explanation which is confirmed by analytical methods. That electro-osmotic transport of water is the essential factor in the impregnation process is indicated by the fact that wood can be electrically impregnated with acid fuchsine, which is carried towards the cathode although it is a negatively-charged colloid. In this case the rate of water transport and the current both increase during the process, and it is concluded that the increase in the negative charge on the wood due to adsorption of the dye has a much greater effect in increasing the rate of water transport than the rising conductivity has in reducing it. Experiments with ferric chloride, mercuric chloride, and copper sulphate show that impregnation occurs much more readily in the sap wood than in the heart, the ratio of concentrations being little different from that obtained by simple diffusion under gravity (Boucherie process), when, moreover, impregnation occurs more rapidly than by the electrical method. H. J. T. Ellingham.

Patents.

Cement manufacture. M. Chatagnier (F.P. 608,658, 8.4.25).—Finely-ground limestone, silica, alumina, and ferric oxide, in the proportions required to produce cement, are mixed with low-grade coal dust in such proportions that the carbon content is sufficient to combine with the carbon dioxide set free from the limestone. The mixture is pressed into blocks and introduced into a hot shaft or rotary kiln. When the formation of carbon monoxide occurs, no more fuel is required, and it is only necessary to supply sufficient air for the combustion of the carbon monoxide. B. W. Clarke.

Cement composition. R. S. Lindstrom (U.S.P. 1,633,790, 28.6.27. Appl., 31.5.24).—Portland cement, ground so that 90% will pass 200-mesh, is mixed with water and with 5—10% by wt. of an iron-free abrasive, of substantially the hardness and sp. gr. of carborundum, ground to 100-mesh. B. W. Clarke.

Manufacture of oxychloride cement. W. J. McCaughey, Assr. to Cleveland Trust Co. (U.S.P. 1,634,505, 5.7.27. Appl., 1.5.22).—An oxychloride cement consists of a comminuted mixture of magnesian limestone in which substantially all the magnesium carbonate is calcined and all the calcium carbonate uncalcined, together with additional magnesium oxide and an alkaline-earth chloride. B. W. Clarke.

Manufacture of plaster material. O. Gerlach (U.S.P. 1,634,459, 5.7.27. Appl., 13.3.25).—Hot, freshly burnt, crushed limestone after being treated with about 20% of its weight of sulphuric acid (*d* 1·71) is mixed and pulverised. B. W. Clarke.

Controlling the rate of setting of hydraulic binding materials. Skanska Cement-Aktiebolaget (F.P. 609,768, 22.1.26. Conv., 26.1.25).—Salts which accelerate the setting of hydraulic binding materials, *e.g.*, calcium chloride, are protected from absorbing moisture by mixing with a colloidal solution, *e.g.*, silicic acid, and evaporating to the required degree of moisture. The product is then ground with the hydraulic binding material. Portland cement or other siliceous binding material, neutralised by hydrochloric acid, may be mixed with the calcium chloride, together with lime or gypsum if required, and the whole evaporated to the desired degree. B. W. Clarke.

Production of material to be used in the formation of roadways etc. E. B. Hack (E.P. 258,870, 20.9.26. Conv., 23.9.25).—Heated bitumen is treated

with a small quantity of a material, such as a solution or emulsion of an oleate or stearate of an alkali metal, casein, etc., which lowers the surface tension between bitumen and steam and causes the evolution of bubbles in the bitumen. The frothy mass is then mixed with a suitable aggregate for use as a road material.

B. W. CLARKE.

Coloured materials for use in the manufacture of hard tennis courts and the like. W. H. W. IDRIS (E.P. 272,582, 10.3.26).—A colouring matter, *e.g.*, green oxide of chromium, with a flux of a relatively low m.p., *e.g.*, lead or boron flux, when burnt at 800° on the surface of sand, crushed brick, or other filler, forms a suitable material. B. W. CLARKE.

Manufacture of cement. E. C. ECKEL (E.P. 268,736, 14.2.27. Appl., 31.3.26).—See U.S.P. 1,591,662; B., 1926, 918.

Production of cement (G.P. 439,154).—See X.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Rusting of steel surfaces in contact. G. A. TOMLINSON (Proc. Roy. Soc., 1927, A **115**, 472—483).—A series of experiments have been made involving a plane surface of hard steel and a spherical surface of steel, glass, agate, or stellite, in relative motion at their point of contact, with the object of discovering the cause of the rusting which occurs at the common surface. The effect of varying the pressure between the surfaces and the condition of the surfaces was examined. The corrosion between chemically-clean surfaces—*i.e.*, surfaces from which any primary film has presumably been removed—is much greater than that between mechanically-cleaned surfaces, even under very low pressures (0·23 g.). It thus seems improbable that the oxidation can be due to any coarse abrasive action between the surfaces. No oxidation occurs, even under very high pressures, unless there is relative tangential motion between the surfaces; hence the effect is not due to the local application of heavy pressure which might conceivably set up an electrolytic action. It is suggested that the effect is a result of molecular cohesion, and experimental evidence is adduced in support of this view. It is found that extremely minute tangential displacements, down to about $6 \cdot 5 \times 10^{-8}$ in. (thus, of the order of magnitude of the radius of molecular attraction), are sufficient to cause the characteristic rusting. Below this value it appears that the molecules may be disturbed and yet return to their original configuration. Experiments are recorded in which steel and glass surfaces were covered with a film of vaseline or castor oil. Oxidation occurred under high normal stresses, when presumably the film was ruptured, but not under low. L. L. BIRCUMSHAW.

Resistance to corrosion of steel containing copper. M. GRISON and E. LEPAGE (Rev. Mét., 1927, **24**, 331—336).—Addition of 0·5—0·7% Cu to mild steel made by the basic or acid open-hearth process increases its resistance to corrosion very greatly. Thus, in 20% sulphuric acid three samples of ordinary mild steel lost 38% in weight after 6 days, whereas similar steels containing 0·5—0·7% Cu lost only 0·9%. In sea water, after 16 days, the losses were 2·3% and 0·35%, in running water 0·14% and 0·065%, and after two months in the atmosphere 0·18% and 0·05%, respectively. The addition of copper had practically no action on the mechanical properties of the metal.

A. R. POWELL.

Comparative tests on ball-bearing steels. T. L. ROBINSON (Trans. Amer. Soc. Steel Treat., 1927, **11**, 607—619).—Endurance tests were made on chromium, chromium–molybdenum, and chromium–vanadium ball-bearing steels similarly hardened and tempered. The results showed wide variations in different heats, steels with marked segregation of carbides giving low values. Even distribution of small cementite particles in the martensitic ground mass was necessary for the highest results in fatigue and static bending tests. The optimum results were obtained on chromium–vanadium steel.

T. H. BURNHAM.

Correlation of magnetic properties with mechanical hardness in cold-worked metal. S. R. WILLIAMS (Trans. Amer. Soc. Steel Treat., 1927, **11**, 885—898).—The scleroscope hardness of 11 cold-rolled nickel strips increased progressively as the thickness decreased. The change in length in a magnetic field decreased asymptotically to the axis of hardness and as the alinement of the crystals became complete. No initial lengthening was observed, and it was concluded that there is also no Villari reversal point for nickel. The area of the hysteresis curves taken on the strips increased with increasing hardness. The specific resistance first decreased and subsequently increased with the hardness. The mechanism for change of length must have some factor distinctly different from that which produces the other magnetic phenomena.

T. H. BURNHAM.

Effect of tempering on the decomposition of austenite. R. L. DOWDWELL and O. E. HARDER (Trans. Amer. Soc. Steel Treat., 1927, **11**, 583—606).—The range of temperature of the decomposition of austenite was 150—200° for high-carbon, carbon–chromium, and high-speed steels, 410—480° for manganese steel, 525—562° for cobalt–chromium steel, and 625—670° for high-nickel steel. It was always higher than the decomposition temperature for martensite in the same steels. In carbon steels the martensite needles darkened progressively at 100°. Micro-examination and sp. gr. tests showed that precipitation of carbide particles took place slowly until very dark troostite was formed. In the alloy steels the martensitic structures decomposed similarly on tempering, but more slowly or at a higher temperature. On tempering austenitic cobalt–chromium and manganese steels, martensitic needles were produced, so that there may be overlapping of the ranges of stability of austenite and martensite in steels of certain composition. Nodular troostite usually forms on tempering austenite, either at the grain boundaries, along slip planes or martensite, cementite, or troostite patches.

T. H. BURNHAM.

Industrial utilisation of aluminium alloys. H. POMMERENKE and P. HERMAN (Rev. Mét., 1927, **24**, 297—306).—For most industrial purposes, especially for motor-car parts, the most satisfactory aluminium alloy is that containing 8% Cu together with not more

than 3% of impurities such as zinc, lead, iron, silicon, and tin. The strongest castings are obtained by melting the alloy below 700° and casting at 675°, whereby a homogeneous fine structure is obtained with a hardness of 70 and a tensile strength of 15 kg./mm.² The best heat treatment of this alloy consists in annealing for 3 hrs. at 500°, quenching in cold water, and ageing at 190° for 1 hr. The Brinell hardness should then be 130 and the tensile strength about 30 kg./mm.² Overheating the alloy during melting results in coarse-grained weak castings which cannot be successfully heat-treated.

A. R. Powell.

Determination of small quantities of zinc in pure aluminium. W. Bohm (Z. anal. Chem., 1927, **71**, 243—246).—When the metal is dissolved in sodium hydroxide most of the zinc goes into solution also and may be separated by addition of sodium sulphide and determined in the usual way. Varying proportions of the zinc, however, remain with the insoluble residue; the whole is brought into solution with bromine and hydrochloric acid, and the copper, zinc, and iron are separated.

S. I. Levy.

Determination of alkalis in ores, clays, and refractory materials. J. Ciochina (Z. anal. Chem., 1927, **71**, 45).—The finely-powdered substance is heated in a steel, copper, or nickel boat in a silica tube for 2 hrs. at 500—600° in a current of hydrogen sulphide. The product is leached with hot water and the residue again treated as above. The combined filtrates from both treatments are saturated with carbon dioxide and boiled for 1—2 min. to precipitate calcium and magnesium carbonates. After cooling, an excess of iodine is added followed by 30 c.c. of 1 : 3 hydrochloric acid. The iodine excess is then titrated with thiosulphate. The iodine consumption is a measure of the sodium or potassium sulphide or both formed during the heating; from these figures the alkali content is readily calculated.

A. R. Powell.

Microdokimastic method [for the determination of the precious metals in ores]. II. G. Lunde (Mikrochem., 1927, **5**, 102—119).—Fuller details are given of the method of determining the precious metals in 1—2 g. of ore by micro-analysis involving measurement of the diameter of the precious metal bead (cf. B., 1927, 302). The separation of silver from platinum by heating the metal bead in boron trioxide leaves a porous particle of platinum unsuitable for measurement, but by again cupelling this together with a known quantity of gold and measuring the diameter of the resulting bead, the weight of platinum present is easily calculated. For determining the precious metals in very dilute solutions a small amount of lead acetate is added and the liquid saturated with hydrogen sulphide. The precipitate is collected, ignited in an unglazed porcelain crucible, and cupelled therein with lead oxide and borax.

A. R. Powell.

Microstructure of the path of fatigue failure in a specimen of Armco iron. F. F. Lucas (Trans. Amer. Soc. Steel Treat., 1927, **11**, 531—549).

Chromium plating. D. H. Killeffer (Ind. Eng. Chem., 1927, **19**, 773—776).

Patents.

Manufacture of cast iron with fine distribution of graphite. Gelsenkirchener Bergwerks A.-G. (E.P. 247,941, 9.2.26. Conv., 21.2.25).—Molten cast iron is heated at a temperature higher than that at which a maximum amount of combined carbon is formed on cooling. When heated at such a temperature, usually 300—550° higher than the solidification point, carbon separates to a large extent in the form of finely-divided graphite on subsequent cooling in chill moulds.

C. A. King.

Simultaneous production of cement and pig iron in the blast furnace. Hochofenwerk Lübeck A.-G. Abt. Rolandshütte (G.P. 439,154, 20.8.25).—Materials containing manganese or phosphorus are added to the charge in order to obtain a freely flowing pig iron rich in manganese or phosphorus. A. R. Powell.

Chilled-iron roll. H. E. Walters, Assr. to United Engineering & Foundry Co. (U.S.P. 1,627,626, 10.5.27. Appl., 23.3.27).—The metal of a cast-iron roll contains 3—3·75% C (total), 0·7—1·25% Cr, 0·15—0·5% Mo, not more than 0·06% P or 0·08% S, 0·75—2·5% Si, 0·2—0·8% Mn. The metal is close grained, and is given a white iron primary chill and also a secondary chill without any distinct line of demarcation.

C. A. King.

[Iron] alloy of high specific electrical resistance unoxidisable at a high temperature. Y. Kamishima (E.P. 272,788, 3.1.27).—An iron alloy containing 6—27% Cr, 0·1—6% Mo, 2—10% Si, and less than 5% of manganese and/or cobalt. F. G. Crosse.

Production of metallic magnesium. M. N. Lacell, Assee. of A. Jessup (E.P. 268,316, 3.3.27. Conv., 26.3.26).—Chlorine produced during the electrolysis of anhydrous magnesium chloride is converted into hydrochloric acid, which is used for dissolving a fresh supply of magnesium oxide or carbonate. The chlorine may be transformed into phosgene with after-decomposition into hydrochloric acid, which may first be used for drying the magnesium chloride produced, or hydrochloric acid may be produced by direct electric or catalytic methods. C. A. King.

Production of metallic magnesium and fixed nitrogen. C. G. Minor, Assr. to Anglo-California Trust Co. (U.S.P. 1,631,544, 7.6.27. Appl., 6.11.22).—Magnesite is dried and calcined in presence of carbon and nitrogen at 1600°, the magnesium nitride formed being then heated with ferrosilicon to give magnesium, which vaporises, and silicon nitride. Alternatively, magnesium oxide is heated with nitrogen and a hydrocarbon, *e.g.*, acetylene, in presence of a high-tension arc to yield magnesium cyanamide, which is treated at about 1200° with an alloy of aluminium and ferrosilicon to form magnesium and aluminium cyanamide, the latter being heated with sodium chloride at 800° to produce aluminium chloride and sodium cyanide.

T. S. Wheeler.

Preventing the oxidation of magnesium and its alloys. I. G. Farbenind. A.-G., Assees. of Chem. Fabr. Griesheim-Elektron (E.P. 254,302, 19.6.26. Conv., 24.6.25).—Oxidation of magnesium is prevented

by treating the surface of the metal with a solution of a fluoride, preferably that of an alkali metal. The metal may then be dried. C. A. King.

Treatment of metal surfaces [against corrosion]. W. Smith (E.P. 272,610, 17.3.26).—As a prevention against corrosion at high temperatures, metals, *e.g.*, aluminium and magnesium, are coated with the sulphide of the metal by exposing the oxidised surface to the action of carbon disulphide vapour in an enclosed chamber maintained at about 700°. C. A. King.

Aluminium alloy. A. L. Mond. From T. Goldschmidt A.-G. (E.P. 272,706, 26.7.26).—In aluminium–magnesium–zinc alloys the relative proportion of magnesium and zinc is such as to form the compound $MgZn_2$, preferably to the extent of 4—12%, but not exceeding 26% of the total alloy. Other elements, *e.g.*, beryllium, silicon, copper, manganese, nickel, titanium, tungsten, molybdenum, capable of forming solid solutions may be introduced. C. A. King.

Aluminium alloys resistant to corrosion especially by sea-water. K. Schmidt (F.P. 608,957, 5.1.26). —Aluminium alloys with up to 12% Mg are made resistant to corrosion by heating the castings at 420° for 5—8 hrs. so as to obtain a homogeneous crystallised mass. Aluminium also forms stable alloys with other metals, such as cobalt, nickel, manganese, and chromium, preferably in the presence of antimony, bismuth, or cadmium owing to the formation of a protective coating on the surface of the alloy. A. R. Powell.

Non-corrodible aluminium[-magnesium] alloy. K. Schmidt (E.P. 272,972, 22.12.25).—The alloy consists of 0·1—4% Mn, 0·05—6% Sb, and 0·1—10% Mg, the remainder being aluminium. F. G. Crosse.

Purifying or refining aluminium and its alloys. D. R. Tullis (E.P. 272,326, 10.5.26).—Dissolved gases and other impurities are removed by introducing chlorine in the form of boron trichloride into molten aluminium. The boron introduced alloys with aluminium and the chlorine forms an equivalent amount of aluminium chloride which rises to the surface of the metal. C. A. King.

Lead alloys. S. Beckinsale and H. Waterhouse (E.P. 272,320, 16.4.26).—A lead alloy not liable to intercrystalline failure and suitable for cable sheathing, pipe, and similar purposes, contains up to 2% Cd, up to 3% Sb, and up to 5% Sn. C. A. King.

[Lead-sodium] alloy. C. A. Kraus and C. C. Callis, Assrs. to Standard Development Co. (U.S.P. 1,628,050, 10.5.27. Appl., 30.3.23).—Molten sodium produced during the electrolysis of a bath of sodium chloride is directly combined in the bath with metallic lead, access of oxygen being prevented. F. G. Crosse.

Production of finely-divided nickel. M. Raney (U.S.P. 1,628,190, 10.5.27. Appl., 14.5.26).—Nickel for catalytic purposes is made by alloying it with aluminium which is subsequently dissolved away. F. G. Crosse.

Extraction of zinc from zinc–iron–siliceous slags. E. Alberti, H. Thielmann, M. Begas, R. and K. Albert (Ver. Werke Dr. R. Alberti & Co.) (E.P. 259,188, 25.8.26. Conv., 29.9.25).—Pulverised slag is mixed intimately with sodium chloride and sulphuric acid and the mixture roasted under oxidising conditions at 450°. Zinc salts are separated from insoluble iron compounds by leaching. C. A. King.

Removal of impurities from minerals and rocks. H. Grunwoldt (E.P. 258,246, 28.8.26. Conv., 11.9.25). —Iron, manganese, etc. are removed from minerals and rocks by treating the granular material with a mixture of sulphuric, hydrochloric, and oxalic acids, under either cold or heated conditions. C. A. King.

Treatment of metal [surfaces prior to electroplating]. Soc. Métallurgique de l'Ariège (F.P. 609,327, 22.4.25).—Before electroplating a metal surface it is pickled in a bath to which a compound of antimony, arsenic, tin, zinc, nickel, phosphorus, sulphur, or copper has been added. The subsequent electroplated coating adheres more strongly and is smoother than one produced on an untreated metal surface. A. R. Powell.

Coating materials [wire] having an iron or steel base. Galvanising apparatus. L. A. Wynd and J. L. Schueler (U.S.P. 1,630,507—8, 31.5.27. Appl., [A], 26.2.26; [B], 20.3.26).—(A) Iron or steel wire is passed first through an annealing furnace, then through an electrolytic cleaning bath containing dilute hydrochloric acid (the wire being made the positive pole and the container the negative), and finally through a coating bath of molten zinc, the surface being kept clean at the points of entrance and exit by regulated addition of ammonium chloride. (B) A zinc coating apparatus comprises a number of small covered pans, each fitted with separate electrical heating apparatus, and with means for supplying ammonium chloride at each end. T. S. Wheeler.

Working refractory metals. F. Koref and K. Moers, Assrs. to General Electric Co. (U.S.P. 1,635,793, 12.7.27. Appl., 29.4.24. Conv., 14.7.23).— See E.P. 218,966; B., 1924, 875.

Metal heating furnace [for rendering iron rustless]. G. Jeffrey (E.P. 272,718, 13.7.26).

Manufacture of pistons from magnesium and its alloys. G. Michel (E.P. 248,720, 10.2.26. Conv., 7.3.25).

XI.—ELECTROTECHNICS.

Electrodeposition of chromium from chromic acid baths. H. E. Haring and W. P. Barrows (U.S. Bur. Stand., Tech. Paper No. 346, 1927, **21**, 413—449). —From a detailed study of the behaviour of a number of chromic acid plating baths it is shown that the essential constituents of these baths are chromic acid and any anion which is not decomposed or precipitated by chromium in any of its valencies, and that, therefore, the so-called acid, neutral, and basic baths which are used commercially are identical both in initial behaviour and in ultimate composition. Maximum efficiency and bright deposits are ensured if the composition of the bath is so maintained that the ratio CrO_3 molarity : SO_4 normality = 50, *i.e.*, if the weight of sulphuric acid is about 1% of that of the chromium trioxide. To this end the chromic acid added from time to time to make up the strength should contain not more than 0·5% of sulphuric acid. For general purposes the bath should

contain 250 g./litre of chromium trioxide and 2·5 g./litre of sulphuric acid or 3·3 g./litre of chromic sulphate. The addition of "chromic chromate" or the formation of this compound in a colloidal form during use decreases the conductivity of the bath and is detrimental to the quality of the deposits obtained; hence the use of chromium or iron anodes, which tends to produce excessive formation of this compound, is not recommended. Lead anodes of large area produce only traces of chromium chromate, and are therefore satisfactory for prolonged use. The best operating conditions for baths of the above composition vary with the nature of the work to be plated, but in all cases the optimum temperature lies between 40° and 60° and the optimum current density midway between that required to give a frosted deposit and that required to give a milky deposit; for nickel or steel this is 16 amp./dm.^{2} and for copper, 25 amp./dm.^{2} The throwing power of the bath is highest for metals upon which the over-voltage of hydrogen is high, so that improved deposits may be obtained on iron, steel, or nickel articles of irregular shape if they are first plated with a thin layer of copper and the circuit is completed immediately the article is placed in the bath. A. R. Powell.

Gas analysis instrument. Griffiths.—See I.

Production of fluorite. Stockbarger.—See VII.

Impregnation of wood by electrical means. Bechhold and Heymann.—See IX.

Quinhydrone electrode. Itano and others.—See XVI.

Alkaloidal titrations. Morton.—See XX.

Patents.

Electric furnace. J. C. Woodson, Assr. to Westinghouse Electric and Manuf. Co. (U.S.P. 1,633,967, 28.6.27. Appl., 1.10.25).—A number of resistors, having alternately similar depending loop portions, are supported by refractory furnace wall members. J. S. G. Thomas.

Electrolytic apparatus more particularly adapted for the electrolysing of alkali-metal chlorides. A. P. H. Dupire (E.P. 267,912, 4.3.27. Conv., 16.3.26).—A stationary anodic bell is provided with an inner peripheric flange on which rest graphite rod anodes and against the underside of which bear the diaphragm and the cathode resting on the cathodic vat carried by horizontal rods adapted to be raised or lowered with reference to the bell by threaded rods. J. S. G. Thomas.

Apparatus for electrolysing solutions of alkali-metal chlorides. E. Krebs (E.P. 264,865, 21.1.27. Conv., 23.1.26).—A permeable cathode is suspended from the horizontal upper edge of the cell casing, and is shaped so as to present active cathode surfaces to both sides of two or more longitudinally extended anodes or rows of anodes arranged side by side across the breadth of the cell. J. S. G. Thomas.

Furnace for electrical treatment of gases for the purpose of oxidation of atmospheric nitrogen or for other purposes. B. Thomas (U.S.P. 1,634,311, 5.7.27. Appl., 14.3.23).—A furnace chamber is provided with tangentially-disposed inlet ports and an outlet port of such size relative to the chamber that a centrifugal motion is imparted to gases passing through the chamber, whereby a low-pressure reaction zone is produced in the region of the operative electrodes. J. S. G. Thomas.

Electrical gas-purifying apparatus. Siemens-Schuckertwerke G.m.b.H., Assees. of C. Hahn (G.P. 438,624, 19.7.22).—Gauze precipitating electrodes of zig-zig, undulatory, or similar cross-section are arranged transversely to the gas stream. J. S. G. Thomas.

Manufacture of a refractory metal product [filament]. C. A. Laise, Assr. to Electron Relay Co. (U.S.P. 1,631,493, 7.6.27. Appl., 18.4.24).—A mixture of tungsten, boron nitride (1—20%), and an alkali metal (less than 1%) is formed into bars and worked at yellow and red heats with intermediate annealings, the diameter of the bars being reduced by hammering and drawing. The wire so formed is heated in oxygen, and the oxide-coated wire further annealed, the oxide being then removed by treatment with sodium hydroxide solution, and the wire drawn down to form electric lamp filaments. T. S. Wheeler.

[Tungsten] alloy filament. A. Pacz, Assr. to General Electric Co. (U.S.P. 1,635,055, 5.7.27. Appl., 15.1.20).—A filament for incandescence lamps consists of tungsten containing 1% Zr. F. G. Crosse.

Insulating and heat non-conducting composition containing mica. T. Okuri (E.P. 272,384, 17.11.26. Conv., 5.8.26).—An electrically insulating and thermally non-conducting material is composed of mica pieces cemented together with a paste of fireclay (1—3 pts.), potassium or sodium borate (20—30 pts.), potassium or sodium silicate (2—5 pts.), magnesium chlorate (0·5—1 pt.), and caustic soda (0·5—1 pt.). J. S. G. Thomas.

Double-fluid cell. L. Darimont (E.P. 266,662, 26.5.26. Addn. to E. P. 241,729).—In a double-fluid cell constructed in accordance with the original patent and with E.P. 250,803 (B., 1926, 551), the exciting solution is composed of a mixture of an exciter proper, *e.g.*, sodium chloride, a caustic material, *e.g.*, caustic potash, etc., an alkaline soap, *e.g.*, potash soap, an albuminous material, *e.g.*, corn flour, a fatty material, *e.g.*, linseed oil, and a mucilaginous or colloidal material, *e.g.*, dextrin, which by reaction with the reagents mentioned produces a fatty membrane capable of opposing diffusion of the alkali metal salts. If desired, chalk and/or kaolin may be added to the exciting solution. J. S. G. Thomas.

Wooden battery separator. E. S. Humboldt, Assr. to Pacific Lumber Co. (U.S.P. 1,631,867, 7.6.27. Appl., 28.8.24).—Green wood slabs of the desired thickness are leached with water, soaked in sodium sulphite or sodium hydrogen sulphite solution at 15° to the b.p. for 5—10 hrs., then washed and finished. T. S. Wheeler.

[Wood separators for] storage battery. W. H. Wood (U.S.P. 1,634,527, 5.7.27. Appl., 15.10.24).—Wood separators for lead accumulators are immersed in sulphuric acid of *d* 1·3—1·5 until the porosity is increased sensibly by removal of cellulose without disintegrating the organic structure of the wood. The concentration of adherent acid is finally reduced. J. S. G. Thomas.

[Supporting sinuous resistors in] electric furnaces. BRITISH THOMSON-HOUSTON Co., LTD., Assees. of C. L. IPSEN and A. N. OTIS (E.P. 259,599, 8.10.26. Conv., 10.10.25).

XII.—FATS; OILS; WAXES.

Stalagmometric tests on some leather oils. W. SCHINDLER (Collegium, 1927, 288—300).—The surface tensions of a fish oil, cold-tested neatsfoot oil, olive oil, and vaseline oil, respectively, increased slightly in a very weak soap or alkali solution, but diminished rapidly with stronger solutions. Tests with these oils showed the surface tension was diminished in the following decreasing order: soap solution, sulphonated fish oil, sodium bicarbonate solution, Turkey-red oil. Egg yolk was only as effective as Turkey-red oil, whilst technical egg yolk had little effect. Increase in temperature diminished the surface tension but slightly. Additions of lactic or citric acid diminished the surface tension appreciably. The formation and stability of emulsions depend not only on the surface tension, but also on the tenacity and elasticity of the aqueous liquid and particularly of the film round the drops; the p_H optima also have an effect. The viscosity of a sulphonated fish oil changes on neutralisation. Hydrophile groups, *e.g.*, CO_2Na, SO_3H, greatly facilitate emulsification. D. WOODROFFE.

Short method for the determination of butter fat. G. D. ELSDON and P. SMITH (Analyst, 1927, **52**, 317—324).—5 g. of the fat are saponified with glycerin and sodium hydroxide, the volatile acids distilled as for the Reichert–Polenske value, but only 100 c.c. of distillate are collected, to which 30 g. of sodium chloride are added, which on dissolution bring the volume up to 110 c.c. After 30 min. at 15° the solution is filtered and 100 c.c. of filtrate are titrated with $0 \cdot 1N$-sodium hydroxide solution to phenolphthalein. The figure obtained, corrected for acidity, if necessary, and multiplied by 1·1 gives the salt-soluble volatile acid figure. The salt-insoluble volatile acid figure is determined as for the Polenske value, except that 18 c.c. of sodium chloride solution (30 g. in 100 c.c. of water) are used instead of water to wash the condenser flask and filter paper. For butter fat the salt-soluble value approximates to the Kirschner value, and the salt-insoluble value is nearly double the Polenske value. To determine the percentage of butter fat (when present in small quantities) in a sample of margarine, the salt-soluble and salt-insoluble values are determined, and the amount of salt-soluble acidity, due to coconut oil and similar oils, is found from the table given, the salt-insoluble figure being taken as due entirely to this source. Approximate values for the salt-soluble and insoluble acidities equivalent to proportions of butter fat in margarine from 0 to 10% are tabulated. D. G. HEWER.

Relations of the Manley and Reichert figures for butter analysis. H. S. SHREWSBURY (Analyst, 1927, **52**, 388—390; cf. B., 1927, 226).—The Manley figures for butter are lower than the Reichert–Meissl by a mean of 15·7% (calculated on the latter value), and it is suggested that this is mainly due to the reactions of soluble fatty acids to methyl orange, accountable from the varying strengths of ionisation of the acids, and in a minor degree to the retention of a small amount of slightly soluble acids by insoluble acids. The possibility of butter fat containing acetic and, particularly, formic acid in the form of glycerides may account for a further difference. D. G. HEWER.

Saponification value of edible fats. J. GROSSFELD and F. WISSEMANN (Z. Unters. Lebensm., 1927, **53**, 244—250).—A new relationship between the saponif. value and the butyric acid number is indicated, and is used as a means of detecting the addition to butter of either coconut oil or margarine. The difference between the saponif. value and 1·5 times the butyric acid number lies between 195 and 200 for most fats, including butter, but excluding coconut oil and margarine, for which the values are 57 and 34 units higher, respectively. Attention is drawn to the precautions necessary for an accurate determination of the saponif. value. 1—2 g. of butter fat are saponified by boiling under reflux with exactly 25 c.c. of $0 \cdot 5N$-alcoholic potash until the liquid clears. The mixture is titrated while still hot in the presence of alcoholic phenolphthalein or Alkali Blue 6B, the latter indicator giving the more readily distinguishable end-point when the titration mixture has a yellow colour. The clearing of the saponification mixture by the disappearance of all droplets of fat does not indicate complete saponification, and it is shown that some fats, especially coconut oil, margarine, and butter fats, require heating for a further 4—8 min. after the clarification, in order to ensure a correct determination of the saponif. value. H. J. DOWDEN.

Alcohol soaps. R. FALCK (Z. angew. Chem., 1927, **40**, 771—776).—Alcoholic soda-coconut oil soaps rubbed well into the skin and then washed to a lather with water afford the best means both for mechanical washing and disinfection of the skin. The effects of varying composition, water content, etc. on the properties of the soap, the rate at which alcohol is lost on exposure etc. are discussed. S. I. LEVY.

Tea-seed oil and its uses as an adulterant of olive oil. H. A. CAULKIN (Pharm. J., 1927, **118**, 769—770).—It is impossible, by any of the tests hitherto suggested, to ascertain whether olive oil is adulterated with tea-seed oil. B. FULLMAN.

Abnormal butter. SCHELLBACH.—See XIX.

Vitamin potency of cod-liver oils. HOLMES and others.—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Protection of steel with paint. A standard paint. J. N. FRIEND (J. Oil & Colour Chem. Assoc., 1927, **10**, 147—163).—As a standard against which protective coatings for iron and steel may be tested, a paint consisting of 80 pts. by wt. of a pure iron oxide pigment and 100 pts. by wt. of pure Calcutta raw linseed oil is recommended. The pigment, dried at 110°, should completely pass 200-mesh and should contain 97—98% of anhydrous iron oxide, the remainder consisting of silica alone or admixed with barium sulphate or calcium sulphate or both. This paint satisfies the requirements for such a standard, *i.e.*, contains the minimum practicable number of ingredients, is easily reproducible over a considerable period, is inexpensively and readily

manufactured, and affords satisfactory protection against corrosion. The details and results of 7 months' immersion tests on this and various other experimental paints are tabulated. In tests against the standard, it is essential that the "hardening periods" both between coats and before immersion be comparable for the various paints under test, and four methods of ensuring this are given. S. S. WOOLF.

Pigment and oil. E. KLUMPP (Farben-Ztg., 1927, **32**, 2306—2307).—A recommended method for determining the oil absorption of a pigment is as follows:—oil is added drop by drop to 1—2 g. of pigment on a glass plate and rubbed in with the tip of the finger until a paste of the consistency of putty is reached, an additional drop of oil permitting the paste to be smeared readily. From a series of oil absorptions determined by this method and from the results of microscopical examination, neither the total pigmentary surface nor particle shape is considered to control the oil absorption. Sp. gr. is the important factor, it being shown that for many pigments the volume of oil absorbed by a given volume of pigment remains constant. The oil absorption of a pigment corresponds to its pore volume at close packing. When, however, a pigment was packed as tightly as possible, and the interstitial volume calculated from the total volume and the known volume of actual pigment, the amount of oil that occupies this space was found to exceed the observed oil absorption, owing to air adsorbed on the particles. Confirmation for this view was obtained by a further series of "bulking" experiments in which the pigment was given a preliminary rubbing with a small amount of Nekal (I.G.), a saponin-like material that assists the removal of adsorbed air layers (and finds use in German grinding practice). The pigment then bulked to an appreciably smaller volume, and calculated and observed oil absorptions agreed within the limits of experimental error. S. S. WOOLF.

Resins. I. Soft, alcohol-soluble Manila copal. P. HORRMANN and N. KROLL (Arch. Pharm., 1927, **265**, 214—226).—Tschirch's work (1900—1902) on copal resins led to the isolation of numerous acids, apparently chemical individuals, but with indefinite characteristics. An authentic sample of soft Manila copal (m.p. *in vacuo*, after drying *in vacuo* at 40°, 251° (decomp.); $[\alpha]_D^{20}$ + 29·6°) yields, when fractionated by repeated extraction of its ethereal solution with sodium carbonate solution of various strengths (as described by Tschirch), acidic substances varying in m.p. and acid value. A better separation is obtained when organic solvents (ether, alcohol, ethyl acetate) are used. From those fractions which have high acid values and m.p., a pure, crystalline, ethylenic, dibasic acid can eventually be obtained. This compound, $C_{20}H_{32}O_4$, is termed *copaldicarboxylic acid*. The *sodium*, *silver*, and *calcium* salts, the *ozonide*, $C_{20}H_{32}O_7$, and the *methyl* ester (b.p. 211°/12 mm.) are described. When the acid is heated above its m.p. carbon dioxide is evolved and the monobasic *acid*, $C_{19}H_{32}O_2$, m.p. 60°, is produced. The *calcium* salt of this monobasic acid is decomposed when heated *in vacuo* and gives an oily *hydrocarbon*, $C_{18}H_{32}$, b.p. 189—191°/12 mm., d^{20} 0·9656. That the raw copal contains about 50% of the dibasic acid is shown by the fact that on dry distillation *in vacuo* it yields both the above hydrocarbon and the monobasic acid in quantities agreeing with that percentage. The raw copal contains also about 12% of ethereal oils and indifferent substances. W. A. SILVESTER.

Plasticised nitrocellulose films.—STEELE.—See V.

Resins in pine. PIGOULEVSKY.—See XX.

PATENTS.

Paints or compositions for protecting substances from the injurious effect of air, water, and other gases or liquids. P. W. TURNER (E.P. 272,142 and 271,912, 1.12.25).—(A) A mixture of kieselguhr in considerable amount and kaolin or similar materials and a white pigment, *e.g.*, titanium or zinc oxide, is ground with a medium consisting of a gum or resin, turpentine, and, if desired, collodion. (B) The surface to be protected is coated with the composition described in (A), and then covered with a suitable fabric or fibrous material, to which the same paint is applied either before or after its application to the surface. S. S. WOOLF.

Reduction of pigment colours and lakes to a finely-divided state. J. W. LEITCH & Co., LTD., and A. E. EVEREST (E.P. 272,585, 11.3.26).—An insoluble soap is precipitated with a pigment colour or lake and the resulting paste is reduced to extreme fineness, *e.g.*, by passage through a colloid mill. On filtering and drying, the pigment or lake retains its finely-divided state. "Soap lake" formation may occur simultaneously with the production of the colour lake. S. S. WOOLF.

Preparation of satin white. J. F. FREDRIKSSON, Assr. to KALBFLEISCH CORP. (U.S.P. 1,632,891, 21.6.27. Appl., 5.2.25).—A mixture of calcium hydroxide, basic aluminium sulphate liquor, and a relatively small amount of water is allowed to react at ordinary room temperature. S. S. WOOLF.

Manufacture of compositions from blood. F. HOMBERG (E.P. 272,380, 3.11.26).—The tough, resistant masses obtained by heating mixtures of blood with white inorganic filling material, *e.g.*, lithopone, zinc oxide, barium sulphate, antimony oxide, etc., under pressure, can readily be bleached by hydrogen peroxide and coloured by suitable dye solutions before or after hardening with formaldehyde. A preferred mixture is 25—60% of filler to 75—40% of blood. If the iron hydroxide arising from the hæmatin be removed by treatment with acid or acid salts, the ivory white tone is corrected and a pure white obtained. S. S. WOOLF.

Coating composition. C. D. HOCKER, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,634,359, 5.7.27. Appl., 6.7.25).—See E.P. 238,696; B., 1925, 890.

Manufacture of white lead. G. F. LLOYD and F. H. CAMPBELL, Assrs. to COMMONWEALTH WHITE LEAD & PAINTS PROPRIETARY, LTD. (U.S.P. 1,635,759, 12.7.27. Appl., 8.8.24. Conv., 10.8.23).—See E.P. 241,329; B., 1925, 999.

Preparation of an ester-resin composition. J. G. E. WRIGHT and W. J. BARTLETT, Assrs. to GENERAL ELECTRIC CO. (U.S.P. 1,634,969, 5.7.27. Appl., 13.6.24).—See E.P. 235,589; B., 1926, 414.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Polymerisation and rubber. J. R. KATZ [with J. SELMANN and L. HEYNE] (Kautschuk, 1927, 215—222). —Stress is laid on the desirability of investigating, for the elucidation of molecular structure, not only the crystalline interference phenomena but also the amorphous rings resulting from the influence of various substances on Röntgen rays. The mathematical relation indicated earlier by Keesom between the diameter of the amorphous ring and the molecular volume does not hold for many polymerised olefinic substances, *e.g.*, metastyrene, polymerised tung oil, and synthetic rubber; for each of these the diameter of the outer amorphous ring is almost identical with that of the unpolymerised substance. From the equality of the values for synthetic rubber and the corresponding butadiene compound it is possible to indicate the chemical origin of a synthetic rubber. Polymerisation, however, sometimes gives rise to a second ring of smaller diameter. On ascending a homologous series, *e.g.*, the *n*-alcohols or *n*-fatty acids, the molecular volume indicated by the amorphous ring approaches a constant value. The indicated molecular volume also approaches more closely to the expected value for a ring compound such as benzene than for a corresponding open-chain, *e.g.*, hexane. Keesom's rule, however, does hold for many substances, and appears to be more exact the more symmetrical or compact the molecule. Although triethylamine gives only one ring the tripropyl, tri*iso*butyl, and tri*iso*amyl bases show two, the inner one of which approximates to Keesom's rule; this is also true of certain ortho-esters and paraldehydes. In such cases the dimensions of the outer ring seem to be associated with the magnitude of the side-chains of the molecule. The results generally indicate that, in many cases where a molecule is constituted from several similar groupings, two amorphous rings occur, the inner one of which agrees with Keesom's rule as to its relation with the molecular volume. With certain colloidal substances obtained by polymerisation, rings of smaller diameter indicative of crystalline constituents of high mol. wt. are observed in addition to the amorphous rings. D. F. TWISS.

Vulcanisation of rubber by sulphur. F. BOIRY (Caoutchouc et Gutta-Percha, 1927, **23**, 13,367—13,368, 13,402—13,403, 13,438—13,440, 13,476—13,477, 13,510—13,511, 13,546—13,548, 13,618).—From a consideration of the features of vulcanisation, particularly in solution, conclusions are drawn that vulcanisation entails (*a*) chemical combination of rubber and sulphur; (*b*) polymerisation of the additive reaction product; (*c*) depolymerisation of the rubber under external influences. The last phenomenon is less marked in vulcanisation at low temperatures with an ultra-accelerator than at higher temperatures. The polymerisation effect follows after the chemical reaction with sulphur, and occurs only between molecules of similar composition and not between sulphur-rich and sulphur-free molecules; the early phenomena of ageing are due in part to continued polymerisation after the vulcanisation operation. The desirable effects of vulcanisation are not due to the sulphur atoms linking together several caoutchouc molecules, but to the greater tendency of the caoutchouc molecules to polymerisation after combination with sulphur. It is probable that in vulcanised rubber all the caoutchouc molecules are not combined with exactly the same proportion of sulphur; the swelling of vulcanised rubber in a solvent is attributed to the portions less rich in combined sulphur. D. F. TWISS.

Vulcanisation of concentrated latex. P. SCHIDROWITZ (Kautschuk, 1927, 202—203).—In the preparation of concentrated latex in a vulcanised condition, the vulcanising agents may be introduced before the concentration operation or subsequently. The presence of an alkali and of a protective colloid is advantageous. By the use of suitable accelerators, vulcanisation may be effected below 100°, although vulcanisation at higher temperatures is possible. The products are of pasty consistency, and can be re-diluted to form vulcanised latex or evaporated to yield vulcanised rubber of attractive mechanical quality. D. F. TWISS.

Determination of moisture in raw rubber. D. ARMSTRONG and T. J. DRAKELEY (Analyst, 1927, **52**, 338—339).—Assuming that results obtained in a vacuum desiccator are correct, drying at 95° in carbon dioxide is recommended when a more rapid method is required, but results will be almost 2% too high, and still more so if hydrogen or coal gas is used. D. G. HEWER.

PATENTS.

Manufacture of thermoplastic derivatives of rubber. B. F. GOODRICH Co., Assees. of H. GRAY (E.P. 260,637, 2.11.26. Conv., 2.11.25. Addn. to E.P. 249,172; B., 1926, 453).—Heat-plastic products are obtained by the action of an organic sulphonyl chloride *e.g.*, *p*-toluenesulphonyl chloride, on comminuted vulcanised rubber scrap or on "reclaimed rubber" at 140° for several hours. Previous admixture of the old rubber with a proportion of new rubber and/or of a softening agent such as rosin is advantageous.
D. F. TWISS.

Treatment of rubber. H. A. BRUSON, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,631,947, 7.6.27. Appl., 28.9.26).—β-Anilinoethyl alcohol is added to a rubber mix to retard the ageing of the vulcanised product. T. S. WHEELER.

Production of surface-finished rubber goods. H. W. EMERY, Assr. to ARCHER RUBBER Co. (U.S.P. 1,631,722, 7.6.27. Appl., 3.11.26).—Rubber is coated with shellac, which is then treated with bromine or sulphur chloride dissolved in carbon tetrachloride and with ammonia gas, to form a tough lustrous surface.
T. S. WHEELER.

Treatment of latex. Centrifugal machine. W. B. WESCOTT (U.S.P. 1,630,411—2, 31.5.27. Appl., [A], 27.6.25; [B], 7.11.25. Renewed 1.12.26).—See E.P. 236,624; B., 1925, 773.

[Metal lining plates for] rubber latex coagulating tanks. SARCO ENGINEERING & TRADING Co., LTD. From W. KELLITT (E.P. 272,608, 17.3.26).

XV.—LEATHER; GLUE.

Tannin of native oaks and true chestnut. A. KURMEIER (Collegium, 1927, 273—288).—The tannin of

oak leaves and twigs (*Quercus sessiliflora*) was isolated. It is amorphous, yellowish-brown, and readily soluble in cold water, methyl and ethyl alcohols, and acetone. It gives a deep blue colour with alcoholic ferric chloride solution. It contains no methoxy-group, shows optical activity, contains about 1% of ash, but neither free nor combined sugars. Hydrolysis with 2*N*-sodium hydroxide at 70° split off 16—17% of ellagic acid, a portion of which is lost owing to the formation of the lactone of hexahydroxydiphenylmonocarboxylic acid. By the action of tannase on the tannin, a product (" quercous acid ") is formed which can be reduced to ellagic acid. The methylated tannin was optically active and had the formula $C_{32}H_{36}O_{16}$. An insoluble dark portion obtained by the methylation had a carbon content 4% higher than in the original material. Dry distillation of the methylated tannin *in vacuo* yielded the trimethyl ester of citric acid, and of the product obtained by methylating the tannin with methyl sulphate yielded citric acid. The yield of pure tannin from chestnut leaves (*Castanea vesca*) was greater than from oak, and yielded 8—9% of ellagic acid. The methylated tannin corresponded to $C_{24}H_{12}O_8(OMe)_8$ and it yielded trimethyl citrate on distillation. The lower molecular weight, the smaller yield of ellagic acid, and the smaller amount of methylated product insoluble in ether indicate that chestnut contains fewer tannins and condensation products of high molecular weight. D. Woodroffe.

Determination of acid in tan liquors by dialysis. L. O. Steven and E. Anacker (Collegium, 1927, 300—301).—10 c.c. of the undiluted tan liquor are pipetted into a diffusion thimble suspended in 150 c.c. of distilled water. After leaving overnight, the dialysate is titrated with 0·1*N*-sodium hydroxide and phenolphthalein. The thimble is suspended in a fresh 150 c.c. of distilled water for 5 hrs., and the dialysate titrated as before. The total acid found by titration should be multiplied by 16/15. No difficulty has been experienced in determining the end-point of the titrations.

D. Woodroffe.

Valuation of gelatin on the basis of its swelling capacity. H. Stadlinger (Chem.-Ztg., 1927, **51**, 507—508).—The results of swelling tests of gelatin of different qualities and thicknesses show that the quality cannot wholly be based on the water absorption, and that the Engler viscosity value affords a better means of determining the suitability of the gelatin. The factors influencing the water absorption are fully discussed. E. H. Sharples.

Leather oils. Schindler.—See XII.

Patent.

Production of material resembling leather. N. Schmitt (G.P. 437,078, 20.3.24).—Mixtures of nitrocellulose, preferably in solution, with a solution of raw or vulcanised rubber in castor oil, or in an ester, acetylation product, or nitration product of ricinoleic acid, with or without the addition of fatty acids insoluble in alcohol, are used. L. A. Coles.

XVI.—AGRICULTURE.

Action of some fertilisers of different physiological and chemical reaction, on the reaction of the soil and on crop yields. O. Lemmermann, L. Fresenius and E. Gerdum (Z. Pflanz. Düng., 1927, **B6**, 241—264).—Fertiliser trials on an acid sandy loam with various salt mixtures are recorded. Best yields of potatoes were obtained when physiologically acid fertiliser mixtures were used. The reverse was the case with barley and sugar beet. In discussion it is shown that the physiological and chemical reaction of fertilisers and the reaction of the soil have important effects on crop yields. The extent of the crop increases is not entirely to be ascribed to changes in soil reaction following the use of fertilisers; and resulting changes in soil permeability would appear to be only of minor importance in this connexion. In all probability the true explanation of these phenomena lies in the chemical changes occurring within the living cell in their relation to external changes in soil reaction. A. G. Pollard.

Lime requirement of acid soils. Slow reappearance of acidity after neutralisation. C. Brioux and J. Pien (Compt. rend., 1927, **184**, 1583—1585).—The slow reappearance of the acidity of soils neutralised by addition of the amount of chalk indicated by the authors' method (cf. B., 1926, 250) is attributed to the formation of organic acids by oxidation of the humus and, also, to the slow neutralisation of slightly soluble silicates.

G. A. C. Gough.

Carbon–nitrogen ratio and microbiological investigation of the soil in rice fields. I. The carbon–nitrogen ratio. A. Itano and S. Drakawa (Ber. Ohara Inst. landw. Forsch., 1927, **3**, 331—336).—Results of a number of fertiliser trials are recorded in which soil reaction, crop yield, and carbon–nitrogen ratio are examined. The ploughing-in of straw without other fertilisers resulted in decreased crops varying with the amount of straw used. Compost and red clover, however, produced increased crops. Generally speaking, soils having wider carbon–nitrogen ratios produced better crops. Although varying amounts of carbonaceous material were used as fertilisers the carbon–nitrogen ratio after cropping was not very different in the various plots. The ratio of the whole series was much narrower than at the beginning of the experiment.

A. G. Pollard.

Biilmann's quinhydrone electrode. III. The electrode and agar bridge. A. Itano, S. Drakawa, and K. Hosoda (Ber. Ohara Inst. landw. Forsch., 1927, **3**, 337—347. Cf. B., 1927, 151).—The quinhydrone electrode was compared with various types of hydrogen electrode and found to give satisfactory results for soil solutions and extracts and various nutrient media used in soil investigations. The agar bridge, with due precaution, can be used repeatedly. Filtered soil extracts have a greater buffer action than soil solutions. The presence of calcium carbonate does not affect the results of p_H determinations by the quinhydrone method.

A. G. Pollard.

Recent researches on soil colloids. A review. C. E. Marshall (J. Agric. Sci., 1927, **17**, 315—332).

Patents.

Preparation of a product [fertiliser] from molasses. W. H. Dickerson, Assr. to Industrial Waste Products Corp. (U.S.P. 1,631,252, 7.6.27. Appl.,

29.12.22).—Molasses, with or without addition of lime, is sprayed into a current of hot gas, in which it is dried and destructively distilled, leaving a charred residue of value as a fertiliser. T. S. WHEELER.

Insecticide. C. A. and S. D. WILKINS (U.S.P. 1,630,506, 31.5.27. Appl., 4.2.24).—An insecticide comprises desiccated molasses, arsenic trioxide, a flavouring, bran, and water. T. S. WHEELER.

XVII.—SUGARS; STARCHES; GUMS.

Properties of corn [maize] starch. Removal of combined fatty acids. T. C. TAYLOR and J. H. WERNTZ (J. Amer. Chem. Soc., 1927, **49**, 1584—1588; cf. B., 1926, 686).—The Rask–Phelps method for the determination of lipoids (B., 1925, 224) removes only a portion of the fatty acids from raw maize starch, and from maize α-amylose. More vigorous application of the aqueous-alcoholic ammonia reagent frees raw and purified maize starch, but not α-amylose, from fatty acids. Electrophoretic separation of the amyloses cannot be effected with fat-free raw starch. It is concluded that the fatty acids are attached to a complex carbohydrate in maize α-amylose, to which they impart a polarity. F. G. WILLSON.

PATENTS.

Centrifugal machine [for sugar]. R. LEHKÝ (U.S.P. 1,628,321, 10.5.27. Appl., 13.5.26).—A centrifugal machine used in the manufacture of *pilé* sugar has a centrifugally-operated receptacle fitted with an upright axle, together with a ring, superimposed on the bottom of the receptacle, which can be raised by means of a series of pins operating underneath by the action of pivoted radial levers. The upper elevation of the axle is also adjustable. J. P. OGILVIE.

Manufacture of starch products and dry starch products. E. STERN (E.P. 272,274, 8.3.26).—A flour of potato, cassava, tapioca, etc. is treated with caustic soda solution and to this is added a solution of barium or aluminium chloride, or a mixture of barium chloride with zinc, copper, or ferric chloride, and alcohol to promote the precipitation. The " metal starch " precipitated is separated, dried in an atmosphere of carbon dioxide to deprive it of at least 25% of its moisture, and then mixed with sodium or potassium sulphate or sodium phosphate or sodium carbonate, with the addition of soap. W. G. CAREY.

Reactivating charcoal (E.P. 264,799).—See II.

Fertiliser from molasses (U.S.P. 1,631,252).—See XVI.

Oxidation of sugars to osones (G.P. 439,115).—See XX.

XVIII.—FERMENTATION INDUSTRIES.

Possibility of protecting wines from secondary fermentations by means of " vaccines " prepared with the aid of cultures of *Saccharomyces ellipsoïdeus* and some cases of apparent inefficacy of this method. P. MALVEZIN (Bull. Soc. Chim., 1927, [iv], **41**, 713—717).—Secondary fermentation of wines can be entirely prevented by inoculation with suitable " vaccines." Such vaccines are obtained by seeding a sweet wine must with a pure yeast at 25° and allowing fermentation to proceed for a time. The temperature is raised to 45° for 1 hr., after which it is lowered to 25° and a fresh amount of yeast added. After 24 hrs. the temperature is again raised to 45° and the treatment repeated until further addition of the yeast produces no fermentation. The mixture is cooled and decanted, the turbid liquid forming the vaccine. The vaccine will not prevent mannitic fermentation, and infection of the wine with this ferment occasions loss of sugar. Fermentation subsequent to vaccine treatment may occur temporarily in wines sweetened with must at 0° and strongly impregnated with sulphur dioxide. It is suggested that the loss of sulphur dioxide consequent upon the rise of temperature reduces the antiseptic condition of the medium and allows fermentation to proceed by the agency of young yeast cells, until on attaining adult age they become susceptible to the toxic action of the vaccine (cf. A., 1926, 867). R. BRIGHTMAN.

Reactions of gallic acid and its presence in fruit and grape wines. J. KLOSS and W. SEIFERT (Oesterr. Chem.-Ztg., 1927, **30**, 117—120).—Gallic acid, with a moderate excess of sodium hydroxide solution, yields a green colour changing to red on addition of sulphuric acid; with Röttgen's reagent (B., 1927, 24) a precipitate is formed, the solution turning green. The former reaction is sensitive to 0·01% of gallic acid, but the change of colour of the latter reagent does not occur with solutions containing less than 0·04%. The ether extract of gallic acid yields a bluish fluorescence with ultra-violet light. Under similar conditions, gallotannic acid gives an orange-red colour with sodium hydroxide solution, a blue colour with Röttgen's reagent, and a blue fluorescence with ultra-violet light. The sodium hydroxide reaction is unsuited to the examination of wines owing to the formation of colouring matters on addition of alkali to the wine. The reaction of fruit wine with Röttgen's reagent is probably due to the presence of gallic acid, which, however, has not been isolated in a pure state from the wine. Of a series of pure grape wines examined with Röttgen's reagent, all but three showed a colour change to green or bluish-green, and these exceptions on addition of 0·05% of gallic acid reacted similarly. The majority of the ether extracts fluoresced with ultra-violet light. The varied behaviour of Röttgen's reagent with grape wines seems to indicate that those which give a green colour contain 0·04% or more of gallic acid, whilst those which remain blue contain less than this amount or none at all.

F. R. ENNOS.

Significance and value of the titration curves of wine. K. TÄUFEL and C. WAGNER (Z. anal. Chem., 1927, **71**, 1—23).—Curves showing the change of p_H of nine different white wines on the addition of hydrochloric acid or of sodium hydroxide have been constructed from electrometric measurements with the quinhydrone electrode; in all cases the curves are characteristic of mixtures of tartaric and lactic acids, and are straight lines up to p_H 4·5, beyond which point they become curved and finally show the characteristic p_H jump at the end-point corresponding with complete neutralisation of the acidity. The p_H value of a white wine may also

be determined by the Bjerrum–Arrhenius double-wedge colorimeter using methyl orange as indicator if the colour of the wine is first allowed for. The slope of the straight-line portion of the titration curve of a wine is a measure of the rate of change of acidity (buffering capacity) on the addition of an alkali or feebly dissociated salt, *i.e.*, $\pi = a/(5\cdot5-p_{H0})$, where π is the buffering capacity, a the titration acidity in mg.-equivalents, and p_{H0} the original hydrogen-ion exponent of the wine. The reduction of the last-named by the addition of a salt of an organic acid may be calculated by means of the equation $dp_H = d_s\ \{1/\pi(1 + K_s/[H^{\cdot}])\}$, where d_s is the number of mols. of salt added to 1 litre of wine, and K_s the first dissociation constant of the salt. Examples of the use of this equation are given, and a method of calculating the p_H value of mixtures of different wines is explained in detail. A. R. Powell.

Sulphur dioxide in malt vinegar. H. E. Cox (Analyst, 1927, **52**, 397—398).—Sulphur dioxide has been found to be normally present in vinegars in quantities varying from 10 to 30 pts. per million (average malt, 12 pts.), and is regarded as derived from the kilning process, addition of finings and caramel, and from sulphured casks. This latter process may impart from 80—130 pts. per million, but the amount tends to be reduced by the fact that the casks may have been wet before sulphuring and afterwards steamed, and also owing to continuous oxidation of the sulphur dioxide. D. G. Hewer.

Patents.

Treatment of yeast for use in the brewing of beer. Hansena A.-G., and L. Nathan (E.P. 271,777, 26.4.26).—Completely sterile storage and treatment of the yeast is obtained by forcing the yeast produced in a primary fermentation directly into a tank maintained at 0—4°, and into which is injected an alkaline regenerating liquid. The yeast is washed by water led through the bottom of the tank and aerated by the passage of air. By means of compressed carbon dioxide, the yeast is transferred to the fermentation vessels from the tank, which is of conical shape at its lower part and furnished with a cooling jacket. C. Ranken.

Fermentation of cellulosic materials. H. Langwell, E. Ricard, and W. A. Burton (E.P. 271,254, 26.11.25).—Fatty acids are produced by the action of thermophilic bacteria on cellulosic materials, the acidity of the mash being maintained within the limits p_H 5 to p_H 9, measured in the bulk of the mash by methods described in E.P. 248,795 (B., 1926, 459). The water and nutrients are wholly or in part supplied by waste liquors, *e.g.*, spent washes from fermentation processes. C. Ranken.

XIX.—FOODS.

Milk products. I. Report of the Milk Products Sub-Comm. to the Standing Comm. on Uniformity of Analytical Methods (Analyst, 1927, **52**, 402—408).—*Determination of total solids of condensed milk (sweetened and unsweetened).*—About 25 g. of prepared sand and a short glass stirring rod are dried to constant weight in a metal dish (aluminium or nickel is suitable), in an oven at 98—100°, and allowed to remain 45 min. in the desiccator before weighing. The sand is tilted to one side of the dish, 1·5 g. of sweetened or 3 g. of unsweetened condensed milk are weighed in, mixed with 5 c.c. of water and then with the sand. The whole, after being heated on a rapidly boiling water bath for 20 min., with stirring in the earlier stages, is transferred to an oven for 1½ hrs. at 98—100°, weighed, reheated for 1 hr., and re-weighed; the loss between successive weighings should not exceed 0·0005 g. *Determination of fat in condensed milk.*—The Röse–Gottlieb method is recommended, using 2—2·5 g. of sample, 8 c.c. of warm water for mixing, 1 c.c. of ammonia solution (*d* 0·88) added to the cooled mixture, and, after mixing, 10 c.c. of alcohol with further mixing. Ether (25 c.c.) is then added, followed, after shaking, by 25 c.c. of light petroleum. After not less than ½ hr. or centrifuging until the solvent layer is perfectly clear, the extract is set aside, further extractions being made with ether and with alcohol, and the clear extracts are mixed. The solvent is then completely removed, the residual fat extracted with repeated washings of light petroleum (allowing any sediment to settle before decantation), and the residue dried at 98—100°. A blank determination should be made with the same quantities of reagents, distilled water being used in place of milk. D. G. Hewer.

Fluorescence of cow's milk in filtered ultra-violet light. O. Gerngross and M. Schulz (Chem.-Ztg., 1927, **51**, 501—503).—Fresh milk of the cow, sheep, and goat shows a strong yellow fluorescence in filtered ultra-violet light. Human milk and the milk of the dog, lioness, mare, and ass shows this fluorescence to a much less marked degree, or else fluoresces blue. In the dairy treatment, the fluorescent material goes over into the skimmed milk, or the butter milk and whey. Cream and butter contain it to only a very small extent, and the proteins freshly separated from the milk and washed with water are free from it. The fluorescent material is only slightly lipoid-soluble. It is resistant to oxidation and to boiling in acid and neutral solution, but not in alkaline solution. The fluorescence disappears in presence of strong alkali (p_H 9), but it reappears again on slight acidification, showing a maximum at about p_H 6, and at p_H 3 it disappears. On subsequent neutralisation it reappears again unaltered. The yellow fluorescence power of milk preserved with formaldehyde lasts for months in the dark. Direct sunlight changes the yellow fluorescence of milk and whey in a short time through white to a bright blue. Diffuse light has the same effect in 24—48 hrs. Simultaneously with this change of fluorescence, bleaching of the normal greenish-yellow colour of the whey by light occurs. W. Clark.

Application of luminescence phenomena to the investigation of impurities in milk. F. M. Litterscheid (Z. Unters. Lebensm., 1927, **53**, 263—264).—Examination by ultra-violet light affords a means of deciding whether the dirt found in milk is due to external contamination or to the milk itself. Dried milk albumin fluoresces from white to yellow with a bluish tinge according to the fat content. The grass, hay, and vegetable residues in cows' excreta do not fluoresce or only dark brown with greenish patches, but when coated with lacquer varnish, the luminescence changes to bright

red. The appearance of milk residues, casein, or albumin curds is not affected by the varnish. The presence of cow excrement is, therefore, readily detected. Fresh green plant sap also exhibits red fluorescence in ultra-violet light. H. J. DOWDEN.

Abnormal butter. H. SCHELLBACH (Z. Unters. Lebensm., 1927, **53**, 267).—A so-called "farm" butter of deep reddish-yellow colour was suspected of having been artificially coloured, but on analysis no trace was found. The Reichert-Meissl number was found to be 34·6, indicating an exceptionally high proportion of soluble fatty acids. Inquiries proved that the cows had been fed principally on carrots and swedes.

H. J. DOWDEN.

Determination of illipê butter in chocolate. H. W. BYWATERS, F. T. MAGGS, and C. J. POOL (Analyst, 1927, **52**, 324—329).—The turbidity temperature of the fat or mixture of fats under examination is determined by placing about 5 g. in a glass tube 6—8 in. long and 1 in. in diameter, closed by a cork through which passes a thermometer, the bulb of which is $\frac{3}{4}$ in. from the bottom of the tube. The tube is inserted in another cork carrying a glass air inlet tube, fitting a 1 litre flask. Air at 18° is aspirated at such a rate as to cause a fall in temperature of the fat of 0·5°/min. The turbidity temperature is that at which the first sign of cloudiness reaches the centre of the bulb, the mean for illipé butter being 30·8°, and for cacao butter 21·4°. Since the turbidity temperature of a mixture of the two fats was found to be proportional to the values for the pure fats, their percentage may be determined. In the case of a mixture of these two fats with a third, provided the third fat can be identified, the proportion of illipé butter may be determined by constructing the curve showing change in turbidity temperature with change in illipé butter content in the presence of the known proportion of the third fat, and then reading off the proportion of illipé butter corresponding to the turbidity temperature of the extracted fat. D. G. HEWER.

Polarimetric determination of starch in marzipan substitutes. A. GRONOVER and E. WOHNLICH (Z. Unters. Lebensm., 1927, **53**, 252—261).—Baumann and Grossfeld's method for the determination of starch (cf. A., 1917, ii, 223) has been applied to marzipan made from apricot kernels. The method is based on the difference between the total rotation of the original material after inversion by boiling with hydrochloric acid and the rotation of the filtrate after precipitation of the starch. Positive results varying from 1·2 to 1·7% of starch were given in all cases where no starch was present. To investigate the discrepancy, the total polarisation of the kernels was measured before and after inversion. When heated, the aqueous extract was clear, but on the addition of hydrochloric acid a faint white precipitate settled out, which was a lævorotatory compound of a protein nature, extractable by water but precipitated by hydrochloric acid, and therefore not present when the total polarisation of the material is determined. In the same way, it was shown that in the water-insoluble residue is present a dextrorotatory substance which is extracted by heating with hydrochloric acid, and appears in the inverted solution from which the total polarisation is determined. When the filtrate is treated with lead acetate, tannin, and sodium sulphate to remove starch, this dextrorotatory compound is also precipitated. In consequence, there is always a positive difference between the two rotations, which may be erroneously regarded as indicating the presence of starch. A similar behaviour was found for sweet almonds. It is concluded that the method is not entirely suitable for the determination of starch in marzipan substitutes, except in so far that any sample which shows an apparent starch content greater than 1·8% may be concluded to contain added starch. H. J. DOWDEN.

Determination of starch in foodstuffs containing meat. V. JAHN (Z. Unters. Lebensm., 1927, **53**, 262—263).—Düring's method (B., 1924, 570) is applied to the determination of starch in sausages, meat salads, or mayonnaise. 20 g. of the material are digested for several hours on a water-bath with 50 c.c. of 8% alcoholic potash, and, after filtering, the residue is washed with hot 96% alcohol, water being added to make a total weight of 25 g. The mixture is treated with 0·5*N*-hydrochloric acid until neutral to phenolphthalein and inverted by heating at 100° for 15 min. after addition of 25 c.c. of hydrochloric acid (80 c.c. of 25% acid diluted to 1 litre). After cooling, 6 c.c. of 4% phosphotungstic acid are added, the whole is diluted to 100 c.c., clarified with kieselguhr, and filtered. The clear filtrate is examined in a polarimeter. The factor 0·475 converts the reading on the sugar scale to % of pure starch.

H. J. DOWDEN.

Sodium selenite [in coffee] as a cause of poisoning. F. RIECHEN (Z. Unters. Lebensm., 1927, **53**, 264—266).—A fatal case of poisoning in which the symptoms suggested arsenical poisoning, proved to be due to the drinking of coffee containing a high concentration of sodium selenite. H. J. DOWDEN.

Vitamin potency of cod-liver oils. XXI. Stimulation of reproduction by fat-soluble vitamins. A. D. HOLMES, A. W. DOOLITTLE, and W. B. MOORE (J. Amer. Pharm. Assoc., 1927, **16**, 518—527).—The influence of fat-soluble vitamins on the reproduction of hens has been examined. Varying amounts of cod-liver oil were added to the food over a period of 32 weeks, the evidence obtained indicating that the addition of fat-soluble vitamin to the diet stimulates egg production, producing eggs of slightly greater average weight and having a more potent vitamin content. The number and viability of the chickens from the oil-fed hens exceeded that of the control birds, which also had a higher mortality. The oil did not impart any detectable flavour to the eggs or to the flesh of the birds.

E. H. SHARPLES.

Detection of mammal bones in fish meal. BARTSCHAT (Chem.-Ztg., 1927, **51**, 518—519).—The meal is passed through 1 mm.-mesh and the coarser portion examined with a microscope for the presence of animal hairs and bones. The finer portion, after extraction of the fat with hot chloroform, is also examined microscopically in a medium of chloral hydrate solution (40 g. of chloral hydrate and 2 g. of water). Evidence of adulteration is also afforded by a tallow-like appearance of the fat, which, in the case

of the pure fish meal, should be clear and fluid. The acidity and refraction of the fat form no safe guide as to the extent of the admixture with foreign bones.

F. R. ENNOS.

Constituents of citrus fruits. S. G. WILLIMOTT and F. WOKES (Pharm. J., 1927, **118**, 770—773).—In the three citrus fruits—orange, lemon, and grape-fruit—the juice contains mostly vitamin-*C*, the grape-fruit rather less than the orange and lemon; vitamin-*B* is concentrated mainly in the flavedo, in considerable quantities; vitamin-*A* is present in significant amounts only in the orange, both in peel and juice. In the ripe fruits, oxygenase is absent; peroxidase is present in flavedo and juice, the concentration being least in orange and highest in grape-fruit. Analytical constants for the rind oils are given. The vitamin-*A* content of cold-expressed ripe orange peel is very high (comparable with some of the most potent animal sources), whilst that of the oil obtained by the acetone–ether extraction of dried grape-fruit peel is very low. The fruit tissue of lemon before development has p_H 6; acidity develops, and by the time of half-growth the p_H is constant at 4 in the peel and 2·2—2·5 in the juice. With ripening, in the orange, there is a steady increase in the contents of reducing sugar and of total sugar. B. FULLMAN.

Less appreciated constituents of orange juice. S. G. WILLIMOTT (Pharm. J., 1927, **118**, 773—775).—The juice of fresh, ripe, Californian oranges contains much more vitamins-*A* and -*B* than was previously suspected (rather more *A* than *B*). Vitamin-*D* appears to be absent. B. FULLMAN.

Determination of sulphur dioxide in dried fruit. J. MILLER (Analyst, 1927, **52**, 338).—1 g. of sodium bicarbonate is dissolved in 300 c.c. of freshly boiled water, 10 c.c. of 20% phosphoric acid are added followed by 32 g. of the dried fruit cut in strips, the mixture being distilled into 5 c.c. of 10 vol. hydrogen peroxide. 150 c.c. of distillate are collected and titrated with 0·1*N*-sodium hydroxide, using methyl orange as indicator, the number of c.c. required multiplied by 100 giving the parts of sulphur dioxide per million. A correction for the acidity of the hydrogen peroxide may be necessary.

D. G. HEWER.

Presence of formaldehyde in wood smoke and in smoked foodstuffs. E. H. CALLOW (Analyst, 1927, **52**, 391—395).—Schryver's method for the determination of formaldehyde (cf. Buchanan and Schryver, B., 1909, 956) was found to be specific for that compound and was used for detecting it in the wood-smoke distillates of oak and deal sawdusts. Confirmation was obtained by preparing the β-naphthol ether from formaldehyde. Wood smoke was found to cause bacon to become impregnated with formaldehyde since a negative Schryver test on the lean of the unsmoked bacon was strongly positive after 2 hrs.' smoking from a freshly prepared laboratory sawdust. Reactions corresponding to concentrations of from 0·5 to 1000 pts. of formaldehyde per million were obtained for purchased smoked foods.

D. G. HEWER.

Examination for halophilic micro-organisms. W. CLAYTON and W. E. GIBBS (Analyst, 1927, **52**, 395—397).—Halophilic cultures require special media for growth, and good growths of chromogenic strains are obtained in 7—14 days with a medium prepared by covering 10 g. of rice grains with 25 c.c. of fish broth and sterilising. The fish broth is made by digesting 1 lb. of minced fresh cod with water, filtering, making up to 1 litre, and adding 0·1% of peptone and 20% of sodium chloride (pure or solar), and adjusting to p_H 8·2. Infection is made with the brine under test and incubation carried out at 37° or at 42° (optimum growth). The presence of chromogenic micro-organisms may be rapidly ascertained by adding the salt crystals directly to the rice medium, when colonies develop where the salt has been in contact with the medium. For non-chromogenic strains, 2% of agar powder and 0·1% of peptone are dissolved in a mixture of fish water and rice water in equal parts by volume; salt is added to 20% concentration, the reaction adjusted to p_H 8·2, and the mixture heated and filtered. With sea water samples, the water (diluted in sterile, 3·5% solar salt solution when necessary) is added to the rice medium.

D. G. HEWER.

Butter analysis. SHREWSBURY. **Butter fat.** ELSDON and SMITH. **Edible fats.** GROSSFELD and WISSEMANN.—See XII.

PATENTS.

Production of concentrated preparations of vitamin-C. L. A. AGOPIAN (E.P. 272,376, 25.10.26. Cf. E.P. 168,903; B., 1923, 470A).—Vegetable juice rich in vitamins is defecated with a mixture of lead and copper acetates *in vacuo* or in an atmosphere of nitrogen, and the filtered juice then neutralised with soda, whereby a precipitate is formed of the glucosides associated with the lead and containing vitamin-*C*. This is twice treated with 5% acetic acid and the solution containing vitamin, after elimination of lead by hydrogen sulphide, is concentrated *in vacuo* and mixed with milk of lime and ethyl alcohol, thus precipitating the calcium salts of organic acids and other impurities, the vitamins in the solution being separated by filtration and the liquid concentrated and dried *in vacuo*. W. G. CAREY.

Production of fish meal. C. SCHLOTTERHOSE (E.P. 272,663, 17.5.26).—Fish material is sterilised in a drum enclosing a conveying device for transporting the mass, the drum being fitted with a jacket for indirectly heating the material with steam at about 105° without admitting moisture or heated air, and is then dried. The bad odours and products of decomposition produced in the sterilisation are withdrawn separately from the concentrated vapours produced during drying, and are then condensed with the drying vapours, any that cannot be condensed being burnt in a furnace. W. G. CAREY.

Dehydration of fruits and vegetables. C. C. MACPHERRAN (E.P. 272,741, 13.10.26).—The material, raised to the dehydrating temperature in a preheating oven, is fed on to a series of belt conveyors travelling in alternate directions, and passes through a countercurrent of hot air. W. G. CAREY.

Heat-treatment of wheaten cereals. R. HUTCHINSON, Assr. to WOODLANDS, LTD. (U.S.P. 1,634,259, 5.7.27. Appl., 26.7.24. Conv., 30.7.23).—See E.P. 228,829; B., 1925, 296.

Manufacture of a yeast stimulant [for bread-making]. J. Takamine, jun., and N. Fujita (U.S.P. 1,634,310, 5.7.27. Appl., 25.7.22).—See E.P. 201,512; B., 1924, 489.

Manufacture of pectin products. D. R. Nanji and F. J. Paton (U.S.P. 1,634,879, 5.7.27. Appl., 15.1.26. Conv., 28.12.25).—See E.P. 269,952; B., 1927, 504.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Capability of the germination of barley as a measure of the toxicity of halogenated hydrocarbons. A. Müller (Chem.-Ztg., 1927, **51**, 510).—Barley grains were treated with the vapour of, and moistened or completely immersed in, a series of halogenated hydrocarbons for 24 hrs. They were then spread out, turned frequently for 24 hrs., and examined for the capability and energy of germination by the usual methods. Contact with the vapour had little deleterious influence on the germination. On the contrary, carbon tetrachloride appeared to stimulate germination slightly. After moistening or completely immersing, the capability of germination was retarded. Tetrachloroethane had the greatest retarding effect, and in decreasing order: pentachloroethane, trichloroethylene, dichloroethylene, carbon tetrachloride, and perchloroethylene. Tetrachloroethane reduced the capability of germination to 62% of that of the untreated material. Examination within periods of 2, 3, and 4 days of the treatment with the liquids showed that the energy and capability of germination were almost completely regained after 4 days. E. H. Sharples.

Synthetic bitter substances of the saccharin-arylsulphonylimide group. A. Klages (with E. Sturm and J. Weniger) (J. pr. Chem., 1927, [ii], **116**, 163—174).—*o*-Toluenesulphonamide (1 mol.) condenses with *o*-benzoicsulphinide ("saccharin"; 1 mol.), yielding o-*benzoicsulphinide*-3-o-*toluenesulphonylimide*, (I), m.p. 255°,

$$\begin{array}{l}C_6H_4\cdot C:N\cdot SO_2\cdot C_6H_4Me\\ \vert \quad\quad \vert \\ SO_2—NH \qquad (I)\end{array}$$

which is also obtained by the oxidation of *o*-toluenesulphonamide with potassium permanganate, by the action of sodium *o*-toluenesulphonamide on 3-chloro-*o*-benzoicsulphinide (ψ-saccharin chloride),

$$\begin{array}{l}C_6H_4\cdot CCl\\ \vert \quad\quad \Vert \\ SO_2—N\end{array}$$

or from ψ-saccharin chloride and *o*-toluenesulphonamide in presence of aluminium chloride. The following salts of (I) are described: *sodium*, $+\frac{1}{2}H_2O$; *potassium*, $+2H_2O$; *calcium*, $+6H_2O$; *copper*, $+\frac{1}{2}H_2O$; *silver*. The silver salt when treated with methyl iodide yields the N-*methyl* derivative, m.p. 128° (*ethyl*, m.p. 155°, *benzyl*, m.p. 165°, and *phenylcarbonato*-, m.p. 225°, derivatives are described). On oxidation with potassium permanganate at 40—50°, I is converted into o-*benzoicsulphinide*-3-o-*carboxyphenylsulphonylimide*, m.p. 190—230° (*disodium* and *silver* salts; *dibenzyl* ester, m.p. 115°). *p*-Toluenesulphonamide and ψ-saccharin chloride yield o-*benzoicsulphinide*-3-p-*toluenesulphonylimide*, m.p. 265° (*benzyl* derivative, m.p. 151°), whilst benzenesulphonamide gives o-*benzoicsulphinide*-3-*benzenesulphonylimide*, m.p. 213° (*benzyl* derivative, m.p. 112°). H. Burton.

Determination of propionic acid in acetic acid. F. Baum (Chem.-Ztg., 1927, **51**, 517—518, 538—539).—12 c.c. of glacial acetic acid or 15 c.c. of 80% acid are weighed into a 100 c.c. flask and diluted to the mark so as to make an approximately 2*N*-solution. 25 c.c. of this solution in a 300 c.c. round-bottomed flask are mixed with 75 c.c. of 0·25*N*-potassium dichromate solution and 70 c.c. of sulphuric acid added slowly. After mixing the liquids, they are heated for 3 hrs., regular boiling being ensured by the addition of capillary glass ebullition tubes, and vapours being condensed by means of a cooling arrangement in the neck of the flask. After cooling, the liquid is diluted to 500 c.c. and 100 c.c. are treated with sodium iodide and hydrochloric acid for determination of the dichromate not reduced. In the reaction, propionic acid is oxidised to acetic acid. If formic acid is also present, it must be determined separately and due allowance made. With pure acetic acid a reduction of the dichromate equivalent to 0·9—1·3% of propionic acid was obtained. With pure propionic acid in small amounts (0·04—0·05 g.) the whole of the acid was determined by this method, but with larger quantities (0·10—0·12 g.) only 87—90% was obtained. Mixtures of the two acids gave similar results, after making allowance for the small amount of oxidation of acetic acid. If the anhydrides of the acids are present, they must be completely saponified with strong sodium hydroxide solution before making up the 2*N*-solution, otherwise the oxidation under the above conditions is incomplete. F. R. Ennos.

Detection of lactic acid in presence of other organic acids. F. G. Germuth (Ind. Eng. Chem., 1927, **19**, 852—853).—Lactic acid and lactates (acidified with a slight excess of hydrochloric acid) give with 15% potassium thiocyanate solution an orange or, with more concentrated solutions, a purple coloration, which is not discharged on addition of a saturated solution of mercuric chloride. The test is not interfered with by the presence of traces of iron, since the coloration produced by ferric salts is readily discharged by mercuric chloride. The test may be used for the detection of lactic acid in the presence of citric acid, tartaric acid, sodium phosphate, sodium phosphite, sodium hypophosphite, sodium succinate, sodium butyrate, sodium benzoate, sodium salicylate, zinc sulphocarbolate, potassium citrate, zinc valerate, sodium potassium tartrate, and ammonium acetate, since these substances give no coloration. W. J. Powell.

Determination of saccharin colorimetrically and by the ammonia process. A. F. Lerrigo and A. L. Williams (Analyst, 1927, **52**, 377—383).—None of the colorimetric methods examined proved adaptable to quantitative work, but the following modification of the ammonia process gave results to an accuracy approaching 10%. The ethereal extract of the acidified sample is evaporated in a 100 c.c. flask and weighed, and a quantity of pure saccharin rather less than this is weighed into a similar flask, 25 c.c. of approximately 3*N*-hydrochloric acid being added to each. After heating the flasks on a steam-bath for 2 hrs., they are cooled and 3*N*-sodium hydroxide solution is added till the contents are alkaline to litmus, the solutions being then made up to 100 c.c. and aliquot portions nesslerised. D. G. Hewer.

Absorption of sulphur dioxide by organic liquids. G. Weissenberger and H. Hadwiger (Z. angew. Chem., 1927, **40**, 734—736).—The following values are the number of c.c. of sulphur dioxide absorbed at 20° by 1 mol. volume (*M.V.*) of the solvent indicated: tetrahydronaphthalene, nil; 10% solution of camphor in tetrahydronaphthalene (*M.V.*, 141·1 c.c.), 1001·81; decahydronaphthalene (*M.V.*, 157·5 c.c.), 1181·25; *cyclo*hexanone (*M.V.*, 103·24 c.c.), 2353·87; 10% solution of camphor in *cyclo*hexanone (*M.V.*, 111·67 c.c.), 1697·38; 20% solution of camphor in *cyclo*hexanone (*M.V.*, 120·09 c.c.), 1765; *m*-methyl*cyclo*hexanol (*M.V.*, 124·6 c.c.), 2082·82; 10% solution of camphor in *m*-methyl*cyclo*hexanol (*M.V.*, 130·9 c.c.), 2264·57; 20% solution of camphor in *m*-methyl-*cyclo*hexanol (*M.V.*, 137·18 c.c.), 1508·98. Compounds containing carbonyl or hydroxyl groups absorb sulphur dioxide to form actual compounds, *e.g.*, : $CO + SO_2 = :C\begin{matrix} \diagup S-O \\ \diagdown O-O \end{matrix}$ Solutions of sulphur dioxide in camphor–tetrahydronaphthalene mixtures are turbid and separate on being kept into two distinct layers, the upper layer consisting of the almost pure hydrocarbon and the lower of camphor and sulphur dioxide. In presence of small amounts of mercury, camphor–*cyclo*hexanone mixtures can take up about 80 times their own volume of sulphur dioxide. Hydroterpene and oil of turpentine absorb 14·3—14·8 and 8·3 times their own volume of sulphur dioxide, respectively, at 20°. In both cases the liquid darkens considerably and a brown substance finally settles out.

J. S. Carter.

Evaluation of digitalis preparations. F. Uhlmann (Arch. exp. Path. Pharm., 1927, **122**, 219—227).—A method of standardising digitalis preparations is given, which consists essentially in that the material is injected intravenously into the abdominal veins. The diastatic time arrest of a dose of 0·005 c.c. of a solution of ouabain, 1 : 10,000 (7 min. on the average), is fixed as unit activity and with it the more important digitalis preparations on the market, as well as other heart remedies, have been compared. W. Robson.

Colorimetric assay of digitalis. L. W. Rowe (J. Amer. Pharm. Assoc., 1927, **16**, 510—516).—Nearly 100 samples of digitalis preparations have been examined by the picric acid colorimetric method under different conditions and with various standards. Of the five standards employed, tincture of strophanthus was the most satisfactory, but high and erratic results were obtained. The frog-heart method of Houghton is considered to be superior to the colorimetric and the Hatcher cat methods, which indicate total activity and do not consider the alimentary absorbable activity-factors not always proportional. The importance of this absorption factor is recognised in the frog method (cf. Wible, B., 1926, 896). E. H. Sharples.

Locating the end-point in alkaloidal titrations. C. Morton (Pharm. J., 1927, **118**, 761—763).—For locating the end-point of alkaloidal and other titrations which can be followed electrometrically, a method is described in which the *E.M.F.* to be measured is applied to the grid of a 3-electrode valve, a potentiometer not being required. The equivalence point is located with an accuracy equal to that of the potentiometric method, measurable deflections of the galvanometer being produced by increments of 0·01 c.c. of the reagent. The galvanometer deflections give directly the $\Delta E/\Delta V$ values (where E = voltage of the cell and V = volume of reagent added) for the construction of the differential titration curve of Hostetter and Roberts (cf. B., 1919, 880 A). B. Fullman.

Technical production of yohimbine. F. Chemnitius (Chem.-Ztg., 1927, **51**, 469—471).—The production of yohimbine hydrochloride, nitrate, and lactate from yohimbine bark is described. An extraction apparatus is sketched for the production of 10 kg. of pure hydrochloride in a yield of 96% from the bark. The reactions and properties of the hydrochloride and a method for the determination of yohimbine in the bark are given.

E. H. Sharples.

Determination of free mercury in commercial products. H. B. Dunnicliff and K. Lal (Analyst., 1927, **52**, 329—332).—Volumetric methods of determining free mercury in commercial products are considered unreliable. Evaporation of an alcoholic mercuric bromide solution gives low results owing to volatilisation of some mercuric bromide, but reduction of mercuric bromide to metallic mercury by alkaline hydrogen peroxide and titration of the halide by Volhard's method was fairly satisfactory. The method proposed is first to remove the main constituents of the material containing mercury by a suitable solvent and then to treat the residue with bromine water, the mercuric bromide formed being dissolved in alcohol, precipitated as sulphide, collected, and weighed in a Gooch crucible. D. G. Hewer.

Identification of pharmaceutically important organic acids and their derivatives. C. A. Rojahn and F. Struffmann (Arch. Pharm., 1927, **265**, 288—307).—A scheme for qualitatively analysing pharmaceutical preparations is described, giving (i) tables drawn up on the "genealogical" principle for carrying out the analysis, and (ii) a list of 32 acids most commonly occurring in such preparations. Under each acid are tabulated those properties best suited for identifying it in following the given scheme. W. A. Silvester.

Essential oils (Bull. Imp. Inst., 1927, **25**, 107—120).—The first six oils investigated are from South Africa and the last two from Seychelles. *Pelargonium oil.* Yield, 0·04% of the fresh herb; d_{15}^{15} 0·901; α_D −8·1°; n_D^{20} 1·466; acid value, 0·8; ester value, 2·0; esters as geranyl acetate, 0·8%; ester value after acetylation, 222·8; total alcohols (as geraniol), 73·6%; a clear solution in 70% alcohol could not be obtained, probably due to the separation of a stearoptene. *Rosemary oil.* Yield of oil, 0·52% of the fresh branches and 1·2—2·0% of the sun-dried material; d_{15}^{15} 0·900; α_D +5·0° at 25°; n_D^{20} 1·469; acid value, 0·9; ester value, 3·0; esters as bornyl acetate, 1·0%; ester value after acetylation, 28·3; total alcohols (as borneol), 7·9%; slight turbidity with 11 volumes of 80% alcohol at 15°. Oil of *Pittosporum undulatum* fruits. Yield of oil, 0·6% of the ripe fruits; d_{15}^{15} 0·8682; α_D +83·5°; n_D^{20} 1·4795; acid value, 1·4; ester value, 14·6; ester value after

acetylation, 47·1; insoluble in 10 vols. of 80% alcohol; solubility in 90% alcohol at 15°, 1 in 5·3 vols. The oil contains about 75% of low-boiling terpenes. *Fennel seed oil.* Three samples are described, (*a*) and (*b*) from South Africa and (*c*) from Southern Rhodesia. Yield of oil, (*a*) 4·5%, (*b*) 6·5%, (*c*) 5·4%; d_{15}^{15} (*a*) 0·977, (*b*) 0·976, (*c*) 0·967; α_D (*a*) +15·4° at 18°, (*b*) +11·3° at 20°, (*c*) +21·6°; n_D^{20} (*a*) 1·536, (*b*) 1·542, (*c*) 1·526; congealing pt. (*a*) 7·5°, (*b*) 10°, (*c*) 2°; m.p. (*a*) 9·5°, (*b*) 12·8°, (*c*) 4°. Sample (*a*) contained 17·5% of fenchone and approximately 56% of anethole. *Dill seed oil.* Yield of oil, 2·6% from the crushed seed; d_{15}^{15} 0·913; α_D +75°; n_D^{20} 1·488; carvone (by normal sulphite method), 56%; solubility in 90% alcohol at 15°, 1 in 0·25 vol. *Palmarosa oil.* Two samples, d_{15}^{15} 0·902, 0·890; α_D +0·3°, nil; n_D^{20} 1·477, 1·473; acid value, 4·2, —; ester value, 54·6, —; ester value after acetylation, 263·1, 266·8; total acetylisable constituents (as geraniol), 90·1%, 91·6%; solubility in 90% alcohol at 15°, 1 in 1·6 vols., 1 in 1·5 vols. *Gingergrass oil.* d_{15}^{15} 0·942; α_D +25·0°; n_D^{20} 1·492; acid value, 3·4; ester value, 10·4; ester value after acetylation, 156·6; total acetylisable constituents (as geraniol), 48·8%; solubility in 80% alcohol, 1 in 2·4 vols. E. H. SHARPLES.

[Roumanian] apricot kernel oil. (MISS) J. MIRCESCU (Bul. Soc. Chim. România, 1926, 8, 28—32).—The oil obtained from Roumanian apricot kernels (58% on dry kernels separated from shells) has the following constants: d_4^{10} 0·9205, d_4^{15} 0·9176, d_4^{25} 0·9117, d_4^{50} 0·8954; n_D^{25} 1·4691, n_D^{40} 1·4640; m.p. —22° to — 4°; acid value, 0·55; saponif. value, 192·8; iodine value, 99·7. The oil has the following composition: total glycerol (8·85%), liquid fatty acids, m.p. — 10° to 0° (74·04%), solid fatty acids, m.p. 22 — 33° (14·34%), unsaponifiable matter (4·34%), and probably contains a large proportion of olein. H. BURTON.

Chenopodium oil from Mauritius (Bull. Imp. Inst., 1927, **25**, 120—122).—Distillation of the leaves and seeds of *Chenopodium ambrosioides* gave 1·4% of oil having: d_{25}^{25} 0·9591; α_D^{25} (100 mm. tube) —3·0°; n^{20} 1·478; solubility in 70% alcohol at 25°, 1 in 13 vols.; ascaridole (U.S.P. method) 61·8%; (Paget's method, cf. B., 1926, 462) 59·7%. E. H. SHARPLES.

Determination of total alcohols in citronella oil. J. DUPONT and L. LABAUNE (Chim. et Ind., 1927, 17, 905—908).—The acetylation of citronellal under varying conditions has been examined. Acetylation according to Schimmel's method but without sodium acetate gave a value of about 50% for esters calculated as *iso*pulegyl acetate, but with quantities of sodium acetate of 0·2—2·0 g. for 10 c.c. each of citronellal and acetic anhydride the quantity of ester formed was proportional to the amount of sodium acetate used, 2 g. corresponding with a maximum ester percentage. This behaviour of the sodium acetate is ascribed to the formation of the additive compound, $(AcO)_2CMe\cdot ONa$ (cf. Higley, A., 1907, i, 461), which is more reactive towards hydroxylic compounds than acetic anhydride. It is concluded that the inconsistency of the results in the acetylation of citronella oil is due to the formation of a mixture of the enolic acetate and diacetate of citronellal. These substances possess very different refractive indices and are easily transformed into each other. Attempts to determine the conditions which limit the formation of one or the other of these forms or which lead to the formation of a definite and constant mixture were unsuccessful (cf. Reclaire and Spoelstra, B., 1927, 427). E. H. SHARPLES.

Relation between the essential oils and the resins [in pine]. G. W. PIGOULEVSKY (J. Russ. Phys. Chem. Soc., 1927, **59**, 299—303).—Experiments were made to determine the mechanism of formation of essential oils and resins in pine, their respective amounts at the various stages of growth of the plant being investigated. It is suggested that the hydrocarbons and resin acids may be formed as decomposition products of the complex esters of trihydric alcohols present. The quantitative relationship that should theoretically exist between the hydrocarbon and the resin acid upon this assumption is confirmed by the analyses of resins from *Pinus silvestris*, *Abies sibirica*, *P. cembra*, and *P. strobus*. J. KAYE.

Micro-analytical practice. MEIXNER and KRÖCKER.—See I.

Acids of wood acetic acid. SEIB. **Hydrocarbons and alcohols from water-gas.** SMITH.—See II.

Gallic acid. KLOSS and SEIFERT.—See XVIII.

PATENTS.

Preparation of emulsions. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 247,588, 11.2.26. Conv., 11.2.25).—As emulsifying agents are claimed salts or free acids of sulphonated, alkylated, and aralkylated or arylated, aromatic or partly hydrogenated aromatic hydrocarbons, excluding diarylethanes obtainable from styrene. Examples are sulphonated benzylbutylnaphthalene, phenylbutylnaphthalene (from naphthalenesulphonic acid, chlorobenzene, and butyl alcohol), benzylamylbutylnaphthalene, benzylbutyltetralene, benzyl*cyclo*hexylnaphthalene, and the product of sulphonating a mixture of oleic acid, savonette oil, naphthenic acids, and aromatic hydrocarbons. C. HOLLINS.

Manufacture of alkylated or aralkylated aromatic sulphonic acids. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 253,118, 2.6.26. Conv., 2.6.25).—By using chlorosulphonic acid or oleum as condensing and sulphonating agent, aromatic hydrocarbons (especially naphthalene) may be condensed even with methyl or ethyl alcohol. Further condensation, simultaneously or subsequently, with higher alcohols or alkyl or aralkyl halides improves the wetting-out properties of the products. The alcohol, naphthalene, and chlorosulphonic acid or oleum are mixed below the b.p. of the alcohol, then heated at 110—140° until completely soluble in water. C. HOLLINS.

Manufacture of esters from aldehydes. C. O. YOUNG, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,630,593, 31.5.27. Appl., 12.8.25).—An anhydrous aldehyde, *e.g.*, acetaldehyde, is treated in the cold with 2—10% of aluminium ethylene glycoloxide monoethyl ether, to yield the corresponding ester, *e.g.*, ethyl acetate, in 85—90% yield. T. S. WHEELER.

Catalytic preparation of alcohols and of other oxygenated organic compounds. L. Casale (E.P. 252,713, 21.5.26. Conv., 28.5.25).—In the reduction of carbon monoxide or dioxide by hydrogen or a gaseous hydrocarbon under pressure to methyl and ethyl alcohols, etc., the mixture of gases before reaching the catalyst is preheated first by means of a heat exchanger in which heat from the reacted gases is utilised, and finally by an electrical device. An arrangement of concentric cylindrical tubes round a central electrical heater by which this principle is carried into effect, is described and illustrated. C. Hollins.

Production of salts of aminoguanidine or ω-aminoalkylaminoguanidines. M. Heyn (E.P. 272,686, 18.6.26).—Alkylenediamines containing less than 6 carbon atoms are made to react with salts of alkyl*iso*thiocarbamides. Agmatine sulphate (ω-aminobutylaminoguanidine sulphate) is obtained in 60—80% yield by adding a concentrated aqueous solution of tetramethylenediamine (putrescine) to ethyl- or methyl-*iso*thiocarbamide sulphate at 100° and evaporating *in vacuo*. Pentamethylenediamine behaves similarly. C. Hollins.

Manufacture of disubstituted guanidines. W. Scott, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,630,769, 31.5.27. Appl., 15.2.23).—A thiocarbamide, *e.g.*, thiocarbanilide, is heated in an autoclave under pressure at 65—70° with aqueous or aqueous-alcoholic ammonia and a basic salt of lead, zinc, cadmium, or tin, *e.g.*, basic lead carbonate, to give the corresponding guanidine, *e.g.*, diphenylguanidine. T. S. Wheeler.

Manufacture of products from aromatic amines and sulphur chloride. J. V. Meigs, Assr. to C. Ellis (U.S.P. 1,631,280, 7.6.27. Appl., 10.5.19).—Aniline or other aromatic amine is treated in the cold with sulphur chloride preferably in a solvent, *e.g.*, benzene, to give coloured pasty products, which, according to the conditions of the condensation, are soluble in dilute acid or in organic solvents. T. S. Wheeler.

Production [separation] of hexamethylenetetramine and ammonium chloride. C. B. Carter, Assr. to S. Karpen & Bros. (U.S.P. 1,630,782, 31.5.27. Appl., 30.7.26. Cf. U.S.P. 1,566,820—2; B., 1926, 217).—A saturated solution of ammonium chloride and hexamethylenetetramine is heated under pressure with ammonia and methylene chloride at 100° for 5—10 hrs., and, after treatment with excess of ammonia, the mixture of ammonium chloride and hexamethylenetetramine precipitated is leached with a number of other batch products on the counter-current system with saturated ammonium chloride solution, thus removing hexamethylenetetramine from the mixture. The solution obtained is treated with ammonia to precipitate hexamethylenetetramine, and the precipitate with that from other batches is leached on the counter-current system with ammonia solution to remove the last traces of ammonium chloride. T. S. Wheeler.

Oxidation of sugars to osones. Chem. Fabr. auf Actien (vorm. E. Schering), Assees. of A. Kraisy (G.P. 439,115, 4.4.24).—Nutrients suitable for diabetic subjects are prepared by adding hydrogen peroxide slowly to acidified sugar solutions containing catalysts, *e.g.*, 3% hydrogen peroxide solution is added to lævulose solution containing sulphuric acid and ferrous sulphate; the product is stirred with calcium carbonate and filtered, and the solution is evaporated to a syrup containing 90% of solid material. L. A. Coles.

Manufacture of solutions of diaminodihydroxyarsenobenzene derivatives. S. R. MacEwan (E.P. 272,567, 17.12.25).—Solutions of arsenical drugs (arsphenamine, neoarsphenamine, sulpharsphenamine, etc.) are stabilised by addition of a sugar and a soluble salt of an acid of phosphorus, *e.g.*, 20 g. of dextrose and 0·5 g. of sodium dihydrogen phosphate for a solution of 5 g. of diaminodihydroxyarsenobenzene formaldehyde-sulphoxylate derivative in 100 c.c. of water. For subcutaneous injection 12 g. of dextrose, 0·5 g. of the acid phosphate, and 3 c.c. of *N*-sodium hydroxide are added to a solution of 5 g. of sulpharsphenamine in 20 c.c. of water. C. Hollins.

Obtaining the active constituents of germ glands. Chem. Fabr. auf. Actien (vorm. E. Schering) (E.P. 261,356, 26.10.26. Conv., 12.11.25).—The extracts of the dried active germ glands are saponified, after determining their saponif. value, preferably by heating with potassium or sodium hydroxide. The unsaponified matter is removed by a solvent (ether). The residue after evaporation of the solvent is purified (especially from cholesterol) by dissolution in ether, filtration and evaporation, and dissolution of the residue in alcohol. The cholesterol separates on concentration and cooling, the hormones being obtained on evaporation in almost quantitative yield. B. Fullman.

Preparation of means for fighting bacterial diseases. C. Räth (E.P. 247,965, 17.2.26. Conv., 18.2.25).—When chemical compounds of the pyridine, quinoline, or *iso*quinoline series (*e.g.*, arsenical derivatives, but excluding alkaloids), which have curative properties in certain diseases, are introduced into a healthy animal, anti-substances are generated in which the applied chemicals are contained in a non-poisonous and highly active state. The anti-substances, which are drawn off from the glands or spleen, are regarded as group-inhibitives reacting against a variety of disease-causing agents, as contrasted with the specificity of antitoxin sera. Both group-inhibitive and specific immunising materials may be obtained, separately or together, by introduction of the chemical compounds and attenuated or virulent cultures into the same animal. Pyridine derivatives containing a negative group in position 2 are very suitable. C. Hollins.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Action of matt-salt (acid ammonium fluoride) on gelatin films. F. Limmer (Chem.-Ztg., 1927, **51**, 503).—By the use of matt-salt, gelatin films are stripped and transferred. Transfer of the image in its original size can be readily carried out, so long as it is not necessary to keep absolutely to the size of the original. The film can be made to extend regularly in the process and enlarged negatives and positives can thus be obtained.

Film extension naturally results in a decrease in density. The extension of the film is dependent on the time of action of the solution; after-treatment of the stripped film; concentration of the matt-solution; the quality of the salt used; the temperature; the nature and condition (age, degree of tanning, thickness, etc.) of the plate film; and the presence of accelerating or retarding substances in the solution. W. CLARK.

Insolubilisation of gelatin of photographic plates by developers. A. and L. LUMIÈRE and A. SEYEWETZ (Bull. Soc. Franç. Phot., 1927, **14**, 102—108).—In the development of exposed plates with pyrogallol, the hardening of the gelatin increases with decreasing sulphite content of the developer for constant carbonate content, and with constant sulphite content it increases somewhat with increasing carbonate. Most organic developers, like pyrogallol, harden the gelatin during development in the parts where there is reduction of the silver bromide, but in the presence of only a very small amount of alkali sulphite, less than 2 g./litre, together with an alkali carbonate, and up to 4 g./litre, in presence of caustic alkali. In the case of pyrogallol, these amounts can extend to 16 g. with alkali carbonate, and to 6 g. with pyrocatechol and caustic alkali. The property of hardening gelatin is common to the oxidation products of the developers studied, with the exception of iconyl (hydroxyphenylglycine). These compounds, formed by the action of bromine on the developer, are destroyed, or are not formed, in presence of sulphite, except in the case of pyrogallol. Certain developers, only little oxidised by air in alkaline solution, such as pyrocatechol, quinol, metol–quinol, and metoquinone, can be used in presence of a small amount of sulphite, to replace pyrogallol for obtaining images by "development" with hot water, as in the carbon process. The use of developers which completely harden the gelatin layer could have interesting application in the preparation of developers for hot countries. W. CLARK.

Developers for hot countries. A. and L. LUMIÈRE and A. SEYEWETZ (Bull. Soc. Franç. Phot., 1927, **14**, 108—110).—Developers containing caustic alkali can be used in hot climates, without disturbing the gelatin, by making use of the hardening action of the products of oxidation of the developer, if the sulphite content of the developer is kept very low. A suitable metol–quinol formula for use at 35—40° contains:—Water, to 1 litre; anhydrous sodium sulphite, 1·5 g.; metol, 1·5 g.; quinol, 1·5 g.; anhydrous sodium carbonate, 10 g.; potassium bromide (10%), 30 c.c. At 30° the time of development is about 2½ min. W. CLARK.

Sodium and potassium carbonates in the developer. LÜPPO-CRAMER (Phot. Rundsch., 1927, **64**, 202—204).—In the case of a metol–quinol developer on full development, the difference between the action of the carbonates of potassium and sodium is very small. In the case of quinol, however, the difference in the effects of the two carbonates is very marked. The greater rapidity of the developer containing potassium carbonate is especially marked at the beginning of development, and although it gets less with increasing time of development, in all normal development times it remains superior. Other slow-acting developers, *e.g.*, pyrocatechol, adurol, pyrogallol, and glycin, show the superiority of potassium carbonate, although not to so marked an extent as quinol. W. CLARK.

Desensitisers. KUHN (Phot. Rundsch., 1927, **64**, 161—163).—The more quinol a metol-quinol developer contains, the less suitable is it for desensitisation with Pinacryptol Green. Other developers, especially neol, are also unsuitable. Very good results are obtained with rodinal and Pinacryptol Green, and this developer is the best for panchromatic plates with desensitisation. Glycin is also good. Thinly-coated plates are in general more rapidly desensitised than thickly-coated plates. 1 pt. of Pinacryptol Green in 20,000 of developer is sufficient. With rodinal, desensitisation by a preliminary bath has no advantages. Pinacryptol yellow, the most powerful desensitiser, can only be used as a preliminary bath. For colour-screen plates, it is better to use a previous bath of Pinacryptol Yellow than Pinacryptol Green, owing to the possibility of green stain with this latter. The advantages of desensitisation are discussed. W. CLARK.

Mercury printing. A. STEIGMAN (Phot. Rundsch., 1927, **64**, 163—164).—Printing with mercury salts is recommended as a substitute for platinum printing. The mercury process is very sensitive to the type of paper employed. For an unsized paper, the following formula is recommended: mercuric chloride, 8 g.; sodium ferrioxalate, 5 g.; tartaric acid solution (15%), 5 c.c.; potassium chlorate solution (5%), 10 c.c. For weakly-sized papers, the following is used: mercuric chloride, 6 g.; sodium ferrioxalate, 1·5 g.; oxalic acid, 0·5 g.; potassium chlorate solution (5%), 5 c.c.; or without the last two constituents. The paper must be sensitised in weak light. With a clear sky in the sun, exposure is from ½—2 min., according to the negative. The developer consists of: (*A*) anhydrous sodium sulphite, 20 g.; mercuric chloride, 1 g.; potassium bromide, 1 g.; water, 100 g.; (*B*) water, 100 g.; metol, 2 g.; anhydrous sodium sulphite, 10 g.; sodium carbonate (cryst.), 1 g. After development, toning is necessary, and Senol (selenium), used in five times the concentration proposed for gaslight prints, is advised. W. CLARK.

Latent image. J. EGGERT and J. REITSTÖTTER (Z. wiss. Phot., 1927, **24**, 350—360).—Earlier experiments indicated that Methylene Blue can act as a nucleus for the formation of fog, but that it cannot act as a catalyst for development (cf. B., 1925, 478). This is quite different from the effect of a pre-exposure on an undyed emulsion. If a plate coated with undyed emulsion is first exposed and then dyed with Methylene Blue, there is the usual fogging action, but the threshold value is not changed. The same result is obtained if physical development before fixing is used. It is concluded that Methylene Blue acts only on silver in the nascent state, and not on silver nuclei which have already been formed. The action is actually the same as that which takes place when desensitisers are used before development, the only difference being that a desensitiser is used which has no fogging action. The action of Methylene Blue may be due to its oxidising power. Nascent silver would be oxidised and so cannot

be formed, whilst nuclei once formed would not be oxidised. From this it would follow that the nuclei are formed by coagulation, since they are less reactive. It must further follow that, on exposure, the silver atom set free must find its way to, or be set free at, the speck of Methylene Blue on the surface of the grain. By the term "coagulation theory of the latent image," the authors mean (1) the silver atom can be formed at a different place from where the light quantum is absorbed; or (2) at preference points on the grain surface there is a gathering of the silver atoms, such places being where certain foreign substances are already adsorbed.

W. Clark.

Time-effect in bleach-out reactions. Lüppo-Cramer (Z. wiss. Phot., 1927, **24**, 380—384).—The latent image is destroyed on further exposure in the presence of phenosafranine, but only when potassium bromide is present. It is now found that this reaction is a time effect, bleaching occurring in absence of potassium bromide, if sufficient time is given for the exposure. Pinacryptol Green instead of phenosafranine gives the bleach-out effect for both short and long exposures. This is also observed with Pinacryptol Yellow, Crystal Violet, Fuchsine, Malachite Green, Brilliant Green, and Brilliant Rhoduline Red. With sufficiently long exposure under a blue filter, phenosafranine without potassium bromide also gives the bleaching. The Herschel effect also shows the time-effect. W. Clark.

Contrast of photographic printing paper. Correlation between sensitometric constants of positive materials and the characteristics of the optimal positives and negatives. L. A. Jones (J. Franklin Inst., 1927, **204**, 41—89; cf. B., 1926, 996).—The theory of tone reproduction is reviewed and applied to a special case. Data suitable for use in correlation work, derived by statistical and sensitometric methods, are tabulated, and the precision of correlation is discussed. W. Clark.

Photographic exposure meter and photometer. F. H. Norton (J. Opt. Soc. Amer., 1927, **14**, 435—444).—Two forms of a simple and inexpensive photometer for determining photographic exposures are described.

W. E. Downey.

Relation between the specular and the diffuse photographic densities. L. Silberstein and C. Tuttle (J. Opt. Soc. Amer., 1927, **14**, 365—373).

Patents.

Colouring a photographic transparency. A. G. Waddingham, Assr. to Color Cinema Productions, Inc. (U.S.P. 1,633,652, 28.6.27. Appl., 8.5.26).—Double-coated kinematograph film having images recording different colour values of a scene on opposite sides of the film, is coloured by bleaching both sides, washing, treating both sides in a uranium nitrate bath to colour both images red, washing, fixing, washing again, and then treating the images recording the green values with a bath composed of ferrous sulphate, ferric chloride, sulphuric acid, and water. W. Clark.

Colouring the image on photographic plates, films, etc. G. Wheeler (E.P. 271,578, 27.2.26).—The image is toned by a solution of titanous chloride or titanium trichloride after bleaching. Between bleaching and toning the image may be treated with a reducing agent such as sodium bisulphite, and it may be dyed with a suitable dye such as Basic Magenta, Azine Scarlet, Methylene Blue, etc. Images on both sides of the film may be dyed with different colours, and one or both toned with titanium. If the dyed image is subsequently treated with the titanium toning bath, the dye is decolorised in the parts where the silver deposit is highest, and the image is converted into a substantially transparent yellow compound. W. Clark.

Production of kinematograph films in natural colours. E. Maurich (E.P. 272,288, 9.3.26).—A printing device is described for printing superimposed monochrome transparencies to form a single multicoloured film.

W. Clark.

Manufacture of unsensitised films for photographic and kinematographic purposes. T. Bausch, V. Bausch, sen. (F. Schoeller & Bausch), and V. Bausch, jun. (E.P. 260,306, 26.10.26. Conv., 26.10.25).—Flexible transparent sheets are prepared from paper by impregnation with an alcoholic solution of a phenol–aldehyde condensation product, the impregnation being carried so far that a coating of the impregnating material is left on both surfaces of the paper after it has absorbed all it can. It is preferable to add ingredients for hardening the product, suitable substances being harder resins such as shellac, or a colloid which can be subsequently coagulated. W. Clark.

Photoprinting. A. E. White. From Rainbow Photo Reproductions, Inc. (E.P. 271,127, 12.1.26).—Colloid printing plates for photomechanical printing are prepared by exposing a colloid-dichromate layer behind a positive without a screen, and washing out the chromium salts unaltered by light, without removing the parts of the colloid unaffected by exposure. After washing, the plate is treated with a reagent, preferably quinol, which hardens the parts of the film not affected by light, but also hardens to a relatively greater extent the light-affected parts. The whole film is then treated with chrome alum solution, after which it is printed from by using an aqueous solution or suspension of a dye. The dye is taken up by the portions of the film which have been relatively unaffected by light, and is relatively repelled by the parts exposed to light.

W. Clark.

Three-colour photography. L. J. B. Didier (E.P. 272,754, 30.10.26).—In the production of three-colour images on two plates assembled image face to image face, the plate bearing the two images superimposed is formed in a single gelatin coating upon which the coloured component images are produced by the use of two dyes having different penetration properties, the one feeble and the other stronger. Suitable dyes are Carmoisine Red F, which has a feeble penetrating action, and Mikado Golden Yellow, which has a much stronger penetrating action. W. Clark.

Recovery of precious metal [silver] from thiosulphate solutions. W. Traxl (Austr. P. 105,084, 5.1.25).—Silver is recovered from thiosulphate or polythionate solutions by the addition of sodium

or hydrogen sulphide insufficient for complete precipitation. Removal of the remaining silver from the filtrate is effected by treatment with magnesium or aluminium turnings. A. R. POWELL.

Photoprinting. E. F. FLAMMER and H. E. SILLIMAN, Assrs. to RAINBOW PHOTO-REPRODUCTIONS, INC. (U.S.P. 1,634,658—9, 5.7.27. Appl., [A], 2.10.24; [B], 11.1.26. Conv., 26.12.25).—See E.P. 271,127; preceding.

Photographic film. I. G. FARBENIND. A.-G., Assees. of W. LOHÖFER (U.S.P. 1,631,421, 7.6.27. Appl., 3.11.23. Conv., 9.3.23).—See G.P. 406,614; B., 1925, 339.

Antistatic photographic film. J. MARETTE, Assr. to PATHÉ CINEMA (ANC. ÉTABL. PATHÉ FRÈRES) (U.S.P. 1,635,681, 12.7.27. Appl., 30.6.25. Conv., 26.5.25).—See F.P. 611,136; B., 1927, 362.

Manufacture of films for colour kinematography. R. BERTHON (E.P. 264,123. 2.7.26. Conv., 9.1.26).

Colour kinematography. SOC. FRANÇ. DES FILMS HÉRAULT, A. RODDE, and A. BOMBARS (E.P. 257,944, 3.9.26. Conv., 4.9.25).

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Nitroglucoside explosive. R. C. MORAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,630,577—8, 31.5.27. Appl., [A, B], 6.11.25. Cf. U.S.P. 1,583,895; B., 1926, 613).—Low-freezing dynamites comprise mixtures of (A) a nitrated polyhydric alcohol glucoside, *e.g.*, nitrated glyceryl glucoside, or (B) nitrated ethylene glycol glucoside, with a nitrated polyhydric alcohol, *e.g.*, nitroglycerin, and other usual ingredients. The glucoside is dissolved in the polyhydric alcohol, nitration being effected with the usual "nitroglycerin" mixed acid. T. S. WHEELER.

Coal-mining explosive. W. O. SNELLING, Assr. to TROJAN POWDER Co. (U.S.P. 1,631,070, 31.5.27. Appl., 30.10.24).—The explosive contains hydrocellulose (1—20%), nitrostarch (5—25%), ammonium nitrate (5—50%), and other usual ingredients. T. S. WHEELER.

Production of smoke fumes, using hexachloroethane and chlorinated naphthalene derivatives. M. METIVIER (F.P. 613,884, 29.7.25).—A solid mixture of hexachloroethane and/or chlorinated naphthalene with zinc dust and tar or an alkali nitrate, packed in a suitable container, is ignited by means of a mixture of, *e.g.*, aluminium powder and potassium chlorate, or calcium silicide and red lead, or hexachloroethane and zinc dust. L. A. COLES.

XXIII.—SANITATION; WATER PURIFICATION.

Occurrence of hydrogen sulphide in the Lake Washington Ship Canal. E. V. SMITH and T. G. THOMPSON (Ind. Eng. Chem., 1927, **19**, 822—823).—The hydrogen sulphide occurring in the brackish water at the bottom of Lake Union (up to 30 mg./litre) is due to the action of bacteria on the sulphates in the water. The appearance of the hydrogen sulphide is accompanied by elimination of the dissolved oxygen.
A. B. MANNING.

Determination of fæcal impurities in water by the detection of *B. Coli*. F. EGGER and F. MAIER (Pharm. Zentr., 1927, **68**, 401—409).—A review and discussion of the methods for the detection of *B. Coli*.
E. H. SHARPLES.

Clarification of large quantities of liquids, *e.g.*, sewage and industrial waste liquors, by dam- or crater-filtration. E. R. BESEMFELDER (Chem.-Ztg., 1927, **51**, 505—506).—The construction, modifications, and working of the dam-filter are described.
E. H. SHARPLES.

Halophilic micro-organisms. CLAYTON and GIBBS. —See XIX.

PATENTS.

Destruction of animal pests. W. BECK, and DEUTSCHE GOLD- U. SILBER-SCHEIDEANSTALT VORM. RÖSSLER (E.P. 272,570, 5.2.26)—Vermin is exposed to a gas containing (by vol.) at least 0·04% of hydrogen sulphide and 0·5% of carbon monoxide, produced by charging cartridges with sulphur, carbon (partly as carbonaceous matter rich in hydrogen), and sufficient oxidising agent (potassium nitrate) to support combustion without giving rise to sulphur dioxide. A metal powder, *e.g.*, iron, is added to supply heat for the reaction by combination with other components, and compounds which produce smoke or which vaporise unchanged may also be incorporated. W. G. CAREY.

Manufacture of vermin killer. E. DERRÉGIBUS (E.P. 272,656, 28.4.26).—Phenols and cresols are diluted in a suitable fluid with pyridine and naphthalene, with or without material to increase the density, and the mixture is compressed. W. G. CAREY.

Garbage reduction. R. D. MACLAURIN and C. C. SMITH (U.S.P. 1,626,853, 3.5.27. Appl., 3.3.26).—A process to reduce garbage economically and without nuisance comprising removal of free water by passing the garbage through a rotary filter, then dehydration of the green garbage in a direct heat dehydrator (preferably of the rotating cylinder type) from its normal moisture content of about 72% to about 35% of moisture, sorting of the partially dehydrated material, thorough disintegration of the material, then further dehydration from 35% of moisture to about 10% in an indirect heat dehydrator (steam tube drier), extraction of fats, and redrying of the resultant tankage from the extractor in a second indirect heat dehydrator. W. T. LOCKETT.

Precipitating from water material forming boiler scale. H. MANZ (Austr. P. 104,741, 22.9.24).—Water-softening agents, *e.g.*, tannin, are made into a stiff paste with water, mixed with sawdust, and dried. Perforated vessels containing the product are suspended in the water under treatment. L. A. COLES.

Water softener. O. W. JOHNSON, Assr. to WARD-LOVE PUMP CORP. (U.S.P. 1,628,541, 10.5.27. Appl., 1.10.26).—In a base-exchange water softener, a certain amount of brine is reserved at the end of the regenerating operation for passage back through the base-exchange material with the flushing water, to regenerate any mineral in the bed wholly or partially exhausted by the hard water passed with the brine through the bed in the regeneration operation. W. T. LOCKETT.

Clarification of liquids (E.P. 272,130).—See I.

// BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 26, 1927.

I.—GENERAL; PLANT; MACHINERY.

Theory of fine grinding. VI. Diameters of irregularly shaped crushed sand particles lifted by air currents of different speeds and different temperatures. G. MARTIN. **VII. Efficiency of grinding machines and grinding media, with special reference to ball and tube mills.** G. MARTIN, F. B. TURNER, and F. LINSTEAD. **VIII. Variation in sp. gr. of quartz sands on prolonged grinding.** G. MARTIN, W. WATSON, and E. BOWES. (Trans. Ceram. Soc., 1927, 26, 21—33, 34—44, 45—58).—VI. Previous work on this subject (cf. B., 1927, 543) is discussed. Two series of experiments were carried out, the air in the first case being in turbulent flow, and in the second in stream-line flow. The lifting power of the air in stream-line flow was much greater than in turbulent flow; in each case the air exhibited the same laws, but with different constants. For very small particles, Stokes' law is obeyed, the constant depending upon the flow of the gas and upon the shape and nature of the particles. At some larger critical diameter Stokes' law becomes unstable, and is replaced by the parabolic law, $V = k\sqrt{d}$, in which k again probably depends upon the same three factors. The temperature of the gas has a marked influence upon the size of particle supported. The method of calculating the correction for temperature is explained. The diameter and weight of quartz particles lifted by air at speeds varying from zero to 328 ft./sec., and the corresponding mesh apertures of industrial sieves are tabulated. VII. The efficiency of a grinding machine is defined as the work performed in ft.-lb. in increasing the surface of standard Leighton Buzzard sand by 1 sq. ft. The work done in grinding is calculated from the formula: $W = B(S_2 - S_1)$, in which S_1 and S_2 are the original and final surfaces of the powder, and B is a constant measuring the efficiency of the machine (work required to increase the surface by 1 sq. ft.). The most efficient grinding media were $\frac{3}{4}$-in. steel balls and 1-in. steel balls. Flints were very inefficient. Maximum efficiency of a mill is obtained when the surface of the sand exceeds that of the balls by from 100 to 500 times. The optimum conditions regarding speed of revolution, percentage load, and volume of the balls and the charge, and the influence of the size of the machine on the efficiency were investigated. Grinding in a current of air did not appreciably increase the efficiency. VIII. The sp. gr. of standard Leighton Buzzard sand and of 17 grades of crushed sand prepared by air-elutriation were determined by the pyknometer method. The maximum increase in sp. gr. in these grades was about 0·2%; hence they contain no amorphous silica. Reducing the grain-size of quartz from 0·75 mm. to 0·30 mm. has no effect on the sp. gr.; further reduction from 0·30 to 0·03 mm. causes an increase of 0·2% in sp. gr. Still further reduction below 0·03 mm. causes a fall in density of 0·07%. Formation of amorphous silica is, therefore, confined to the grade having a particle size below 0·03 mm. The amount of amorphous silica formed by prolonged grinding varies directly as the time of grinding, after the initial increase in sp. gr. has ceased. The amount of amorphous silica formed after grinding standard sand for $3\frac{1}{2}$ hrs. was 0·91%. F. SALT.

Recovery of volatile materials by condensation. G. WEISSENBERGER (Kautschuk, 1927, 162—163).—Methods for the recovery of volatile liquids such as acetone by cooling are considered. Although self-contained drying systems, such as those with internal or external circulation of the drying gases, enable a greater recovery by condensation than those operated with a continuous draught, they are less desirable on grounds of safety. The yield possible with any condensation process is naturally lower than that attainable with an absorption or adsorption process.

D. F. TWISS.

PATENTS.

Centrifugal separator. V. E. METCALFE (U.S.P. 1,630,201, 24.5.27. Appl., 16.8.23. Renewed 6.4.27).—A rotary conveyor is arranged within and co-axial with an annular rotary screen, and an electro-inductive drive is provided with means for varying the relative speeds of the conveyor and the screen. H. HOLMES.

Apparatus for the cooling or heating of liquids. H. WILKINSON (E.P. 270,537, 18.8.26).—In apparatus of the type where one liquid trickles down over a vertical stack of tubes and the other liquid zig-zags through them, a method is described of making the joints between the tubes and the end connexions so as to give flexibility.

B. M. VENABLES.

Distillation apparatus. W. K. LEWIS, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,626,346, 26.4.27. Appl., 5.12.23).—An apparatus for fractional distillation comprises a still and a fractionating column, and means for leading the vapour passing from the still to the intermediate portion of the column through a coil in the bottom of the latter, to supply the heat necessary for fractionation. T. S. WHEELER.

Apparatus for deodorising fluids. TE AROHA DAIRY Co., LTD., and H. L. MURRAY (E.P. 270,509, 11.6.26).—The hot fluid is admitted through a float

chamber to a closed cylindrical container which communicates through a goose-neck with an ejector-condenser to produce a vacuum. The container is fitted with beaters, and its lower portion is steam-jacketed so that intense ebullition of the liquid is caused, and the vapours are expelled through the ejector. A centrifugal pump discharges deodorised liquid continuously from the container. W. G. CAREY.

Stabilisation of colloidal systems. CONSORT. FÜR ELEKTROCHEM. IND., Assees. of W. O. HERRMANN and W. HAEHNEL (U.S.P. 1,629,161, 17.5.27. Appl., 1.7.26. Conv., 17.7.25).—Sols, *e.g.*, hydrosols containing metals, metallic oxides, metallic sulphides, etc., can be stabilised by addition of polymerised vinyl alcohol, which is obtained as a white powder, soluble in water, by saponifying polymerised vinyl acetate. T. S. WHEELER.

Absorption refrigerator. E. WIRTH, Assr. to SULZER FRÈRES SOC. ANON. (U.S.P. 1,630,648, 31.5.27. Appl., 18.11.24. Conv., 22.12.23).—See E.P. 231,240; B., 1925, 385.

Kiln and other heat-treatment furnace. C. W. SPIERS, Assr. to MORGAN CRUCIBLE CO., LTD. (U.S.P. 1,630,587, 31.5.27. Appl., 21.1.26. Conv., 9.9.25).—See E.P. 261,866; B., 1927, 76.

Determining critical temperature. G. F. GERDTS (U.S.P. 1,630,485, 31.5.27. Appl., 12.12.24. Conv., 26.8.24).—See E.P. 240,636; B., 1925, 945.

Driving device for centrifugal separators. AKTIEBOLAGET SEPARATOR (E.P. 267,842, 19.1.27. Conv., 18.3.26).

[Rotary valve for] pulp thickeners or filters. R. HADDAN. From OLIVER CONTINUOUS FILTER CO. (E.P. 270,616, 11.6.26).

Separation of gases (E.P. 248,377).—See VII.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Netherlands East Indian coals. J. VAN DER WAERDEN (Fuel, 1927, **6**, 252—256).—The coal deposits in the East Indian Archipelago belong to the eocene or younger tertiary formation; they are brown coals of high volatile matter content. Their moisture content is, in general, lower than that of the European brown coals, and their ash content frequently lower also. Some of the coals, *e.g.*, the Lematang, are of bituminous or anthracitic type. Their caking power is small, and is entirely lost on exposure of the coal to air. No positive conclusions with regard to their behaviour on carbonisation, and in particular their capacity to yield a good coke, can be drawn from a comparison with other coals of similar proximate analysis, or from their content of "oil bitumen" and "solid bitumen." The most satisfactory method of utilising these coals would appear to be by blending with coals of high caking power before carbonisation. A. B. MANNING.

Gasifying fuels by [a mixture of] oxygen and steam. T. CERASOLI (Gas- u. Wasserfach, **70**, 508—510).—The methods of calculation used by the author for determining the composition of mixed gas (B., 1924, 4) have been extended to a study of the gasification of carbon by a mixture of oxygen and steam. The oxygen requirement, equilibrium temperature, quantity, and composition of the resulting gas have been calculated for different values of the steam consumption per kg. of carbon. The method has also been applied to the complete gasification of a typical coal by a mixture of oxygen and steam. The gas yield per kg. of coal is equivalent to 6005 kg.-cal. (1·643 $m.^3$ of gross calorific value 3654 kg.-cal./$m.^3$), as compared with 4996 kg.-cal. when air is used, the oxygen requirement being 0·387 $m.^3$ A. B. MANNING.

Reactions in the fuel bed of a gas producer. II. Effect of depth of fuel bed and rate of firing. R. T. HASLAM, R. F. MACKIE, and F. H. REED. **III. Effect of steam-coal ratio.** R. T. HASLAM, J. T. WARD, and R. F. MACKIE (Ind. Eng. Chem., 1927, **19**, 119—124, 141—144).—II. Tests were made in an experimental gas producer burning anthracite to determine the effect of depth of fuel bed and rate of firing on the reactions in the fuel bed. Steam was admitted at a constant rate of 0·4 lb. per 1 lb. of fuel, and samples were drawn through water-cooled sampling tubes from different points in the bed. Runs were made with beds of 1·5, 3·0, and 4·5 ft. depth, with firing rates of 10, 40, and 70 lb. of coal per sq. ft. of grate area per hour. The heating value of the gas, cold gas efficiency, and % of steam decomposed increased with the depth of fuel bed and the rate of firing. With increasing depth of bed and rate of firing, the temperature of the primary reduction zone is increased, as also is the proportion of carbon monoxide and hydrogen in the gas, at the expense of carbon dioxide and steam. The increased time of contact with low rate of firing does not compensate for the resulting low temperature. The thickness of the primary reduction and the oxidation zones is not affected by increase in depth of fuel bed or rate of firing. The combination of oxygen and carbon is independent of the time of contact, but is greatly affected by the velocity of the oxygen past the carbon face. Steam passes through the oxidation zone without appreciable decomposition III. Under the same conditions as above, the effect of increasing the steam per lb. of coal from 0·366 to 1·03 lb. was tested, with a constant depth of fuel bed of 3 ft. and a constant rate of firing of 40 lb./sq. ft. of grate area. The heating value of the gas produced and the cold gas efficiency increased to a maximum, and then decreased, the optimum ratio being 0·7—0·8 lb. of steam to 1 lb. of coal. This value is higher than those obtained by other experimenters, owing to the high rate of firing employed, and the ratio, it appears, increases with the rate of firing. The best results are obtainable from producers by high rates of firing and steam consumption. H. MOORE.

Measurement of bloom of lubricating oils. L. M. HENDERSON and H. C. COWLES, JUN. (Ind. Eng. Chem., 1927, **19**, 74—76).—The bloom was measured by a modified Ives tint photometer, in which a septum prevented the light from the photometer lamp striking the oil, a beam from an auxiliary (75-watt Mazda) lamp being cast on the surface of the oil at 45°. The oil container was a crystallising dish sand-blasted inside and out, and painted black outside. Colours were

reflected from an oil–air interface, and differed from those reflected from oil–glass. The layer of oil was 35 mm. thick and the temperature about 35°. At higher temperatures oils reflect less, but assume their bloom on cooling. The reflected colours of asphalt and white oil are similar. Deblooming increases the absorption of blue light. Debloomed and fatty oils have similar reflected and transmitted colours.

H. Moore.

Determination of oil in paraffin wax: direct refractometer method. S. H. Diggs and C. C. Buchler (Ind. Eng. Chem., 1927, **19**, 125—127).—The indices of refraction for wax-free oil and oil-free wax were determined. Pressed distillate of the lowest cold test obtainable represented the oil component, and had a cold test of — 22°, and n_D^{60} 1·4762. An oil-free wax was obtained by repeated treatment of a wax–oil mixture with filtering clay, and had m.p. 54°, n_D^{60} 1·4361. The wax does not suffer fractionation by filtration, but the values of n of waxes vary with their m.p. The curves of indices of refraction are based on the assumption that the index for 100% of oil constituent is a fixed point, that for 100% of the wax constituent being determined. The curve indicates the percentage of constituents for a certain refractive index, and a chart is given to facilitate its determination. For waxes containing less than 2½% of oil, the index is determined at 60° on crude wax, and then on wax filtered through clay. For waxes with more than 2½% of oil, the index at 60° is alone determined, use being made of the established value for the oil-free wax component. The percentages of oil determined by this method are generally higher than by the press method with waxes containing small amounts of oil.

H. Moore.

Petroleum waxes. C. C. Buchler and G. D. Graves (Ind. Eng. Chem., 1927, **19**, 718—722).—Zaloziecki's theory (B., 1888, 428) that the change from amorphous to crystalline paraffin wax is due to a change from *iso*- to normal paraffins and Gurwitsch's views to the contrary are reviewed. Samples of ordinary paraffin wax, slop wax from higher-boiling distillates, petrolatum from residual stocks, and rod wax from petroleum wells were freed from oil by recrystallising from ethylene dichloride to constant m.p., and were fractionally distilled under a pressure of less than 1 mm. of mercury. That decomposition or rearrangement did not occur was shown by recombining aliquot parts of the fractions and still residuum, when this mixture had in each case the same m.p. and refractive index as the undistilled material. After removal by recrystallisation of a portion designated " soft wax " from all the fractions, a straight-line relationship between refractive indices and m.p. was found to hold, this relationship being identical for all four types of wax. Mol. wts. were determined cryoscopically and confirmed by combustion analyses, and the m.p.–mol. wt. curves were found to be in close agreement with that of Kraft for synthetic normal paraffins. It is concluded that all four types of wax are composed of members of the normal paraffin series ($C_{18}H_{38}$ to $C_{43}H_{88}$), and, as all the purified fractions crystallised in the form of plates of the same type, it is believed that the " soft wax " which was removed is in some way responsible for the needle form of ordinary crystalline wax.

C. O. Harvey.

Mechanism of coking. E. Audibert and L. Delmas (Chim. et Ind., 1927, **17**, 707—722).—See B., 1927, 383.

Bone black. Knowles.—See XVII.

Patents.

Separating coal from dirt and like substances. W. H. Berrisford (E.P. 265,341, 11.12.25).—Coal containing dirt is fed on to the upper of a series of smooth inclined plates which are spaced. The lower end of each plate is turned upwards slightly, whilst an adjustable slide also turned upwards is attached to the upper end of each plate. The coal slides down the plates and leaps from plate to plate, describing a parabolic path. By turning up the lower and upper edges of each plate the paths of the coal and the dirt are made relatively more divergent. The gaps between the successive plates can be easily adjusted so that the dirt falls between the plates and the coal passes forward.

S. Pexton.

Coal washing apparatus. C. Marchant (E.P. 264,460, 22.11.26. Conv., 16.1.26).—Coal is fed into a vertical conduit, where it meets a succession of upward currents of water fed into the conduit at different levels. Corresponding to each water inlet there is an auxiliary conduit branching from the opposite side and at a higher level than the water inlet. As the coal meets the streams of water the lightest fractions are removed into the auxiliary conduits, and only the heavy scale passes through to the bottom.

S. Pexton.

Manufacture of carburetted water-gas. A. E. Shippee, Assr. to Stone & Webster, Inc. (U.S.P. 1,630,316, 31.5.27. Appl., 18.1.21).—In a plant consisting of a generator and a carburettor, a process is operated consisting of three steps—an air-blast run, a steam run, and an independent run of free air through the carburettor.

R. A. A. Taylor.

Producing air-gas from inflammable liquids. H. Foersterling (U.S.P. 1,628,135, 10.5.27. Appl., 5.3.25).—The liquid is vaporised with air in such proportions that the mixture burns without further addition of air, the oxygen content being slightly above that required for complete combustion, further dilution with air rendering the mixture non-combustible.

C. O. Harvey.

Manufacture of air gas. H. Foersterling (U.S.P. 1,624,046, 12.4.27. Appl., 28.3.25).—Air impregnated with a mixture of methyl alcohol and acetone vapour at ordinary temperature contains sufficient combustible gas to combine with all the oxygen present, and can be ignited at a jet to give light and heat.

T. S. Wheeler.

Gas production. T. F. Rintze (F.P. 611,236, 13.2.26).—High-boiling hydrocarbons are cracked, the condensed light products converted by distillation into light or slightly higher-boiling secondary products, and the heavier condensation products vaporised by heating, permanent gases being formed thereby.

A. B. Manning.

Gas generators and the like. Patent Retorts, Ltd., and T. M. Davidson (E.P. 270,429, 12.2.26).—A

retort in which coal is carbonised is superposed on a generator so that the coke which is formed in it may pass straight in to be gasified. A pair of such retorts is arranged so that the hot gas passes down one generator and then up through a flue into the top of the retort on the second generator. The connecting flues may be placed vertically in the setting of the generators and connected with the top and the bottom of the generators by means of radially-disposed passages, or arranged externally to the generators, extending cross-wise from the bottom of one generator to the top of the other retort. R. A. A. Taylor.

Removal of hydrogen sulphide from coal- or water-gas. W. E. Leuchtenberg (U.S.P. 1,629,396, 17.5.27. Appl., 20.7.25).—An alkali carbonate solution is used to absorb hydrogen sulphide from the gas in question. The solution is then revivified by being passed counter-current to a flow of air, the contaminated air being subsequently freed from hydrogen sulphide by passing it through iron oxide moving counter-current to it. R. A. A. Taylor.

Low-temperature carbonisation. G. Mars (G.P. 436,918, 3.10.23).—The material is carbonised in a shaft furnace, the heat being supplied by the combustion of part of the material itself. The position and temperature of the combustion and carbonisation zones can be accurately controlled by suitable regulation of the air supply, which is distributed over the whole cross-section of the shaft. A. B. Manning.

Prevention of sticking of the material in continuous carbonisation processes. I. G. Farbenind. A.-G. (Swiss P. 116,958, 11.11.25).—The material is compressed into briquettes, which are then powdered with the dry material before being charged into the retorts. A. B. Manning.

Working up the volatile products of wood distillation. V. Freund (Austr. P. 104,143, 6.12.24).—The vapours from the distillation of wood are freed from tar and the acetic acid then absorbed by passage through a tube heated externally at 100°, through which at the same time quick or slaked lime is continuously passed. The residual vapours, consisting chiefly of water and wood spirit, are separated by dephlegmation and condensed. Dry, solid calcium acetate and highly concentrated wood spirit are thus obtained directly.

A. B. Manning.

Production of gasoline. H. B. Setzler, Assr. to National Refining Co. (U.S.P. 1,629,810, 24.5.27. Appl., 9.6.16).—Carbon is separated from gas-oil by distilling at 370—400° a volume of the oil, maintained substantially constant and approximately filling the still, at 50—100 lb. pressure. Means are provided for scouring the bottom of the still and for advancing the hydrocarbons to the point of withdrawal during the distillation. H. Holmes.

Refining petroleum. W. E. Lummus (U.S.P. 1,628,252, 10.5.27. Appl., 21.5.23).—The vapours of the lighter fractions of the oil are fractionated by passage through a series of condensers, the uncondensed vapours being absorbed in higher-boiling condensate.

C. O. Harvey.

Treatment of impure petroleum oils. J. D. Brady, Assr. to Brady Process Co. (U.S.P. 1,628,574, 10.5.27. Appl., 29.8.25).—Emulsified petroleum is treated in a vertical tank containing a funnel head and conductor tube down which the oil passes to a chamber containing baffles and heated from below. Valves are provided for the withdrawal of oil and gases.

C. O. Harvey.

Separating metallic base reaction products from other derivatives in the treatment of hydrocarbon oils. Litharge Recovery Corp., Assees. of A. Kinsel (E.P. 263,730, 8.3.26. Conv., 26.12.25).—An apparatus which facilitates the recovery of lead sulphide formed during the treatment of oils with sodium plumbite (cf. U.S.P. 1,525,301; B., 1925, 276, and Kinsel, B., 1926, 37) consists of a steam-heated chamber, into which the alkaline sulphide wash from the agitator is run. Coagulation of the sulphide is effected by heating with or without the addition of coagulants such as ferric sulphate, and any hydrocarbon material volatilised during heating or steam-blowing is condensed and collected. The oily and aqueous alkaline layers which separate in the chamber are drawn off through suitably placed cocks, and the lead sulphide residues pass to a filter, and may be regenerated by treatment with sulphuric acid followed by dissolution of the lead sulphate in a concentrated solution of caustic alkali. C. O. Harvey.

Continuous [bleaching] treatment of oils. O. J. Salisbury (U.S.P. 1,628,747, 17.5.27. Appl., 11.4.24).—Oil is introduced into a treating chamber, to which partially spent fuller's earth is added. After agitation, the oil is passed to filters through a series of treating and settling chambers, in which it is treated with fresh fuller's earth. Means are provided for returning some of the oil carrying partially spent fuller's earth from the settling chambers to the initial treating chamber.

T. S. Wheeler.

Furnace for the treatment of spent fuller's earth. R. E. Wilson, Assr. to Standard Oil Co. (U.S.P. 1,630,044, 24.5.27. Appl., 23.2.24).—An inclined rotary shell is provided with means for internal heating and for supplying fuller's earth into its upper end. An air conduit leading into the shell communicates with a distributing pipe leading from the conduit to the lower side of the shell, and thence close to the wall of the shell in the direction of rotation. The portion of the pipe normally covered by the fuller's earth is perforated.

H. Holmes.

[Cracking] treatment of hydrocarbons. W. S. Hadaway, jun., Assr. to Texas Co. (U.S.P. 1,628,143, 10.5.27. Appl., 15.9.23).—The oil enters a vertical cylindrical cracking chamber through a hollow rotatable spraying mechanism actuated by the oil.

C. O. Harvey.

Conversion of oils. C. P. Dubbs, Assr. to Universal Oil Products Co. (U.S.P. 1,628,236, 10.5.27. Appl., 8.10.21).—Oil, cracked under pressure in a heating zone, passes to an insulated vapour chamber in which a pool of liquid oil collects. The vapours pass to a dephlegmator and condenser, the reflux condensate is returned to the cracking zone, and the uncondensable gases are heated and passed through a conduit immersed

in the oil in the vapour chamber, and are finally discharged into this oil. C. O. Harvey.

Cracking petroleum oil. R. T. Pollock, Assr. to Universal Oil Products Co. (U.S.P. 1,628,270, 10.5.27. Appl., 6.12.20. Renewed 6.10.26).—The oil (under pressure) is cracked, substantially vaporised, and subjected to reflux condensation. The condensate is distilled at atmospheric pressure in a still heated by the flue gases from the furnace of the cracking zone, and the high-boiling still residues are recracked.

C. O. Harvey.

Preparation of bituminous emulsions. Union Franç. de Crédit (F.P. 611,479, 19.2.26).—The molten bitumen is mixed with 3—5% of an animal or vegetable oil, the latter being brought into solution by the addition of a strong acid or an oxidising agent without permitting the temperature to fall; finally a dilute boiling alkali solution, in volume between once and twice that of the bitumen, is added. A. B. Manning.

Manufacture of agglomerated fuel. L. Liais (U.S.P. 1,630,662, 31.5.27. Appl., 5.4.26. Conv., 30.4.25).—See E.P. 251,605; B., 1926, 907.

Motor fuel. J. la Riboisiere (E.P. 257,613, 25.8.26. Conv., 26.8.25).—See U.S.P. 1,557,257; B., 1925, 950.

IV.—DYESTUFFS AND INTERMEDIATES.

Vat dyestuffs of the anthraquinone [pyrazoleanthrone] series. A. Holl, Assr. to Grasselli Dyestuff Corp. (U.S.P. 1,627,738, 10.5.27. Appl., 8.12.25. Conv., 22.12.24).—Pyrazoleanthrone-yellow, or a derivative, is treated with paraformaldehyde in presence of concentrated sulphuric acid, to yield *dyes*, which give on cotton from a hyposulphite vat, reddish- to greenish-yellow shades, fast to light and alkali. The shades obtained can be varied by varying the concentration of the acid and the temperature of condensation.

T. S. Wheeler.

Manufacture of benzanthrone. R. G. Caswell and E. G. Marshall, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,626,392, 26.4.27. Appl., 31.7.20).—Anthraquinone (3 pts.) dissolved in concentrated sulphuric acid (44 pts.) is treated at 38—42° with freshly precipitated copper (2 pts.), and when dissolution of the copper is complete, glycerol (4 pts.) and water (4 pts.) are added, and the mixture is heated at 120° for 3 hrs. The yield of benzanthrone is about 90%.

T. S. Wheeler.

Azo dyes from aminodiphenylene oxide. E. Glietenberg, Assr. to Grasselli Dyestuff Corp. (U.S.P. 1,624,944, 19.4.27. Appl., 2.12.25. Conv., 11.12.24).—Diazotised 3-aminodiphenylene oxide is coupled with 2-naphthol-3-carboxylo-α-naphthylamides to give *dyes* yielding clear claret shades on cotton, which, when directly produced on the fibre, are very fast to light and kier boiling. T. S. Wheeler.

Production of indophenol. J. G. Dinwiddie, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,628,534, 10.5.27. Appl., 18.3.24).—In the production of indophenols by the condensation of derivatives of carbazole with derivatives of nitrosophenol in presence of sulphuric acid, products of increased purity are obtained by performing the condensation at below − 4°.

T. S. Wheeler.

Separation of tertiary from secondary and primary aromatic amines. British Dyestuffs Corp., Ltd., E. H. Rodd, and R. W. Everatt (E.P. 270,930, 21.8.26).—The dry mixture of amines ordinarily obtained by alkylation etc. of arylamines is treated with chlorosulphonic acid, whereby the primary and secondary amines are converted into sulphamic acids. On steam distillation from alkali, the tertiary amine is obtained pure. The sulphamic acids are hydrolysed with dilute sulphuric acid, and secondary and primary amines recovered. A mixture containing only secondary and tertiary amines gives each in pure condition. If the original mixture contains more than 15% of secondary amine, it should be diluted with tertiary amine or a solvent. The separation of mono- and di-ethylanilines (12% of the former, 88% of the latter) is described.

C. Hollins.

Manufacture of dihydroxyperylene. A. Zinke and A. Klingler, Assrs. to F. Bensa (U.S.P. 1,629,194, 17.5.27. Appl., 4.10.22).—See E.P. 191,363; B., 1924, 34.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Provision of cellulose for viscose silk factories and utilisation of waste lyes from the manufacture. F. Giordani and A. Cittadini (Giorn. Chim. Ind. Appl., 1927, 9, 165—167).—The extraction of cellulose by the Italian gaseous chlorine method and the manufacture of viscose artificial silk are conveniently worked in conjunction, the waste lye from the pressing of the alkali-cellulose being used for the preliminary alkali soaking of the vegetable fibre. Under suitable conditions this procedure exerts no deleterious effects on the yield or quality of the viscose silk, and does not increase the amounts of reagents used. T. H. Pope.

Evaporation of sulphite[-cellulose] waste liquor. W. L. Badger (Ind. Eng. Chem., 1927, 19, 677—680).—An evaporator, in which the liquor is mechanically circulated, thus minimising the formation of scale on the heating surfaces, is described. The liquor is pumped through vertical, steam-heated nickel tubes enclosed in a tower, issues from the upper ends of the tubes in the form of spray, and returns to the pump down the sides of the tower. After a run of 150 hrs., during which the test conditions (steam pressure etc.) were adjusted to imitate those in the several stages of a quadruple-effect evaporator, no scale was formed inside the tube, and the heat transfer coefficients were several times as high as those obtained in a standard vertical or horizontal tube evaporator. W. J. Powell.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Patents.

Conditioning [bleaching] textiles. W. L. Conrad (U.S.P. 1,630,786, 31.5.27. Appl., 25.5.23).—The material, with the same end always foremost, is passed in a continuous process through a vat or kier containing a boiling cleansing solution, through a washing machine, a bleaching bath, a piling machine, a washing machine, a hypochlorite or similar bath, a second piling device,

and then through a final washing machine. After leaving each bath, the excess liquor is squeezed out before the material passes to the next, and the latter is submitted to air-bleaching during its passage through the piling devices. B. P. RIDGE.

Reserving animal fibres. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 248,007, 22.2.26. Conv., 20.2.25).—Wool or silk in union fabrics resists direct cotton dyes when certain synthetic tanning materials are applied to the fibre, or are added to the direct dye-bath; two-colour effects are thus obtainable. The substances used are aromatic, or partly hydrogenated aromatic, sulphonic acids condensed through a carbon, sulphur, or oxygen atom with a sulphonated or unsulphonated residue, and suitable examples are sulphonated ditolyl methylene ether, sulphonic acids of benzylated and alkylated naphthalenes, the salts of sulphonic acids obtained by condensing naphthalene with sulphur chloride in sulphuric acid or by oxidation of a sulphurised phenol resin, or by condensing a phenol-aldehyde resin with sulphobenzyl chloride. Thus half-wool, dyed with Naphthol Yellow SEL or Amidonaphthol Red BB, is immersed in a bath containing Dianil Pure Blue PH or Dianil Green BBN and 3% of the reserving substance; or half-silk, dyed with Ponceau G, is immersed in a bath containing Dianil Green GN and 3% of the reserving substance. C. HOLLINS.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Rotary kilns for pyrites burning. C. P. DEBUCH (Papierfabr., 1927, 25, 365—379).—The principle of the rotary kiln has not hitherto been applied to pyrites burning owing to the very large proportion of air required for the reaction and the necessity for close temperature-control if sintering is to be avoided. The Kauffman kiln avoids these difficulties by distributing the air between a number of ports along the kiln. This arrangement incidentally causes the cinders to be discharged at a much higher temperature than usual. To complete the burning off, the latter part of the kiln is fitted with scrapers. These also improve the mixing of the gases and are equivalent to a lengthening of the kiln. They are constructed by the insertion of special projecting blocks in the internal brick lining. The pyrites is charged and the cinders are withdrawn by devices arranged to exclude air-leaks. The air-ports consist of cast-iron sockets, leading well to the interior of the kiln, protected with an outer brick lining and easily removed. They are constructed for either suction or pressure supply, and also serve as observation holes. The gas exit pipe passes downwards through a bend luted into the flue so as to allow for the play necessary consequent on the rotation of the kiln. Earlier forms of this apparatus failed owing to the length being insufficient, but the kiln described has been in constant use since 1924, on pyrites fines. The capacity is 10 tons per 24 hrs. The residue is well burnt; the gases leave at 500—800° and average 10% SO_2. From 4% to 5% of the sulphur used is burnt to sulphur trioxide. The dust produced and the reduction in size of the pyrites particles during combustion do not seem to be very different from corresponding results with a shelf burner. The power required is 5 h.p., and in spite of the need of careful control, labour requirements are on the whole less than with the shelf burner. Whilst high-class constructional materials must be used, the capital outlay does not exceed that for the best shelf burners. The life of the lining of the kiln is estimated at 3—6 years. C. IRWIN.

Calcium nitrate. MCCANDLESS and BURTON.—See XVI.

PATENTS.

Treatment of brine. A. K. SMITH and C. F. PRUTTON, Assrs. to DOW CHEMICAL CO. (U.S.P. 1,627,068, 3.5.27. Appl., 29.9.23).—Brine containing calcium and magnesium chlorides is concentrated to *d* 1·38, and filtered from separated sodium chloride. The filtrate is evaporated under reduced pressure, when tachydrite separates until the greater portion of the magnesium chloride is removed. The precipitate of tachydrite is washed with 25% of water at 22—50°, and with saturated magnesium chloride solution. Relatively pure magnesium chloride remains. T. S. WHEELER.

Manufacture of ammonium nitrate. H. HOWARD, Assr. to GRASSELLI CHEMICAL CO. (U.S.P. 1,625,807, 26.4.27. Appl., 26.5.26).—Air is passed up a tower in counter-current to a descending stream of aqueous ammonia, and the mixture of air and ammonia gas thus obtained is led through ammonium nitrate solution, to which nitric acid is simultaneously added. The air freed from ammonia and water vapour is returned to the tower. The heat of reaction between ammonia and nitric acid is used to concentrate the solution of ammonium nitrate as it is formed. T. S. WHEELER.

Preparation of alkali metal cyanides. R. W. POINDEXTER, JUN., and P. T. DOLLEY, Assrs. to CALIFORNIA CYANIDE CO., INC. (U.S.P. 1,624,147, 12.4.27. Appl., 17.4.26).—Hydrogen cyanide is introduced at about 600° into a molten mixture of sodium carbonate and sodium cyanide, and, when reaction is complete, carbon is added, the mixture being heated at 900° to reduce any sodium cyanate and sodium cyanamide, and to decompose any sodium ferrocyanide present. The product, while still molten, is filtered through a perforated iron plate, and contains about 95% of sodium cyanide. T. S. WHEELER.

Production of alkali hyposulphites. I. G. FARBENIND. A.-G. (Austr. P. 104,397, 19.10.25. Conv., 14.2.25).—Alkali bisulphites are reduced with alkali amalgams under such conditions that the alkali sulphite content of the reaction mixture is kept as low as possible. L. A. COLES.

Production of ammonium polysulphide. D. F. WILHELMI (Dutch P. 15,635, 6.11.24).—Liquid ammonia is stirred with an excess of sulphur heated above its m.p. L. A. COLES.

Manufacture of sodium azide. F. WILCOXON and B. GROTTA, Assrs. to ATLAS POWDER CO. (U.S.P. 1,628,380, 10.5.27. Appl., 27.10.24).—Hydrazine hydrate solution (1 pt., 50% N_2H_4), sodium hydroxide (7·49 pts.) in absolute alcohol (7·5% solution), and ethyl nitrite (1·76 pts.) react to give sodium azide in 90% yield. T. S. WHEELER.

Manufacture of magnesium oxide and calcium pentasulphide. V. Drewsen, Assr. to West Virginia Pulp and Paper Co. (U.S.P. 1,628,311, 10.5.27. Appl., 11.12.23. Renewed 8.10.26).—Dolomitic lime is slaked and the aqueous suspension resulting is heated with sulphur under pressure at 120°. A solution of calcium pentasulphide and thiosulphate is obtained, leaving a residue of magnesium hydroxide.

T. S. Wheeler.

Treatment of alunite. T. A. Mitchell, Assr. to L. M. Hughes (U.S.P. 1,628,174, 10.5.27. Appl., 9.8.22).—Ground alunite (100 pts.) is calcined at 1200° with calcium carbonate (80 pts.), and the product extracted with sodium carbonate solution to give a solution of sodium and potassium aluminates, carbonates, and sulphates, which is treated with carbon dioxide to precipitate aluminium hydroxide, concentrated to separate alkali sulphates, and then returned to the process. In a modified method, by using a mixture of calcium and barium carbonates, no sulphate passes into solution on treatment of the calcine with sodium carbonate solution. T. S. Wheeler.

Production of arsenic compounds. E. R. Rushton (U.S.P. 1,624,281, 12.4.27. Appl., 28.5.24).—Arsenic trioxide vapour is passed with air over calcium oxide at about 900°, when tricalcium orthoarsenate, which is of value as an insecticide, is rapidly formed in satisfactory yield. T. S. Wheeler.

Separation of crystals from solution and the regeneration of heat in connexion therewith. P. H. Müller (E.P. 269,068, 18.10.26).—Heat is recovered from hot solutions or lyes from which salts are deposited on cooling, by passing the hot solution through a series of closed vessels at diminishing pressures, the vapours formed thereby being used to heat liquid for the treatment of further crude salt. The vacuum vessels are connected by horizontal or inclined pipes, and the inlet pipe for hot liquid terminates so far below the liquid level that the pressure at the mouth of the pipe is equal to or greater than the vapour tension of the liquid, whilst downwardly extending pipes are connected with pumps to draw off the salt which settles out, the salt from the first two vessels being removed separately. In the upper part of the vacuum vessels are placed mixing or surface condensers at different levels in each vessel, and joined by siphon pipes through which the cold lye circulates in the reverse direction to the hot liquid passing into the vessels, air being drawn off from these condensers by ejectors actuated by steam or lye. W. G. Carey.

Dispersion of matter into a finely-divided form. H. L. Glaze, Assr. to A. R. Maas Chemical Co. (U.S.P. 1,625,203, 19.4.27. Appl., 30.9.24).—Sulphur (1 pt.) is mixed with soda ash (3 pts.), and the mixture is heated at 120°, cooled, and extracted with water. The sulphur is obtained in a finely-divided form. T. S. Wheeler.

Separation of gases. Ges. für Linde's Eismaschinen A.-G. (E.P. 248,377, 24.2.26. Conv., 25.2.25).—The process applies particularly to the production of mixtures of hydrogen and nitrogen from gases which contain also carbon monoxide and other gases. Partial condensation is effected under pressure by cooling the compressed gases by heat exchange from countercurrent flow. The components less volatile than nitrogen are thus partially liquefied, so that the hydrogen in the residual gas reaches a concentration approximating to that desired. The residual gas is then washed with liquid nitrogen under pressure, whereby the constituents not required are replaced by nitrogen. The washing is effected at nearly constant temperature; the washed gas is then heated by heat exchange with the unwashed gases, and is expanded, the resultant cooling effect being utilised for liquefying nitrogen; or this refrigerating effect may be transferred indirectly to the nitrogen by means of the gas mixture remaining after liquefaction.

R. A. A. Taylor.

VIII.—GLASS; CERAMICS.

Microstructure of earthenware. H. Insley (J. Amer. Ceram. Soc., 1927, **10**, 317—326).—Samples of glazed earthenware bodies, varying in composition and in firing treatment, were examined microscopically. The development of mullite and the solution of the quartz, both within the body and at the contact of the body and glaze, were particularly noted. In general, the greater the heat treatment of the unglazed ware the longer the mullite crystals within the body and at the point of contact of body and glaze. Mullite crystals at the contact grow out from the body into the glaze normally to the plane of contact. Such crystals are, in general, larger than those found within the body.

A. T. Green.

[Terra cotta] colour problem. H. Spurrier (J. Amer. Ceram. Soc., 1927, **10**, 330—333).—Pieces of white enamelled terra cotta became contaminated by a chromium compound during particular kiln firings, the colour being impaired. Microscopic examination of pieces of the enamel which had flaked off from the body showed that the colour effect (pinkish) was entirely superficial, and was located in small spots of rather greater opacity than the contiguous areas. These spots, which were the nuclei for the colour formation, proved to be rich in tin oxide. The coloured enamel contained chromium in amounts greater than those found in the original constituent ingredients. Further, the kiln atmosphere carried chromium. By passing carbon dioxide over heated charcoal in a Hoskins furnace and allowing the products of the reaction to act on the pinkish-coloured ware for a short time in the furnace, the colour was removed. A test for chromium was achieved by the use of a solution of diphenylcarbazide in glacial acetic acid, the solution being made up with methyl alcohol. Use of complementary colour effects is also developed in the detection of these spots.

A. T. Green.

Ageing of enamels. H. G. Wolfram and R. H. Turk (J. Amer. Ceram. Soc., 1927, **10**, 334—338).—The ageing of an enamel as a suspension causes the clay and other particles to assume effective colloidal properties, thus increasing its power of cohesion to the metal base. It is shown that where the necessity for rapid production does not allow time for ageing, soaking of the clay is an excellent substitute. The mobility and yield value of enamel suspensions are discussed. A. T. Green.

Solubility of enamel frit in mill water. H. L. Cook (J. Amer. Ceram. Soc., 1927, **10**, 339—343).—Periodic occurrences of "pinholing" were proved to be due to the fusion of borax crystals formed as the sprayed enamel dried slowly. A 600 lb. batch of enamel was milled and 25 lb. samples were taken at intervals during the grinding. The fineness of the particles was determined and the liquors were analysed. The solubility of the frit increases with increasing fineness. After dry-grinding, the enamel frit is in such a state that an appreciable portion of the soda and boric acid immediately dissolves. Further experiments suggest that the operations of mixing, smelting, puddling, and resmelting have little effect upon the solubility. A. T. Green.

Effect of various electrolytes when added to enamel suspensions made with and without clay. H. L. Cook (J. Amer. Ceram. Soc., 1927, **10**, 344—346).—A clay slip, an enamel suspension, a finely-ground clay-sand slip, and a finely-ground sand suspension were subjected to the action of the electrolytes, magnesium sulphate, magnesium hydroxide, barium chloride, ammonium carbonate, calcium hydroxide, calcium chloride, borax, sodium hydroxide, and sulphuric acid. The anticipation that the "setting-up" or stiffening of water suspensions is dependent on the presence of clay was not realised, for suspensions without clay showed marked action. It is suggested that, in the circumstances of the experiments, the fineness of grinding and degree of solution of the suspended material are factors of greater importance than the colloidal properties of the clay. A. T. Green.

Some dryer considerations. R. S. Troop (Trans. Ceram. Soc., 1926, **25**, 352—360).—A *résumé* of the problems involved in formulating drying schedules and determining the efficiencies of drying plants, together with a consideration of the methods in vogue for drying refractories, clay wares, moulds, and cores. A. T. Green.

Patent.

Composition for coating metal surfaces. R. R. Danielson (U.S.P. 1,629,072, 17.5.27. Appl., 1.12.23. Free of use in U.S.A.).—A mixture of silica (64·9 pts.), borax (36·96 pts.), sodium nitrate (6·95 pts.), and triplumbic tetroxide (8·17 pts.) is sintered at 800—900°, the product being ground and mixed with enamel clay (93 pts.) and water to form a paste which is employed to protect metallic surfaces in selective carburisation. The surfaces are pickled in acid before application of the paste. T. S. Wheeler.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Direct production of iron [from its ores]. F. Wüst (Stahl u. Eisen, 1927, **47**, 905—915, 955—964).—The production of sponge iron from iron ores by the methods of Edwin, Wiberg, Hornsey, and the U.S. Bureau of Mines is discussed at length, and the chemical equilibria and heat-balance of the various methods are compared. Direct reduction with carbon may be effected at temperatures between 700° and the sintering point of the ore without the use of an excess of reducing agent and with the minimum loss of heat energy; the reaction is strongly endothermic, and the reduced metal usually contains more or less phosphorus and sulphur. On the other hand, reduction with carbon monoxide commences at 800—850°, is slightly endothermic, and requires a large excess of gas, entailing greater heat losses, but the product contains relatively little sulphur and phosphorus. In the Edwin process the gases from the reduction are cleaned by passage through a lime-tower, heated at 1600° together with some vaporised oil in a high-tension arc, and regenerated by passage through coke to which sufficient lime is added to slag the ash. The ore is reduced at 800—850° and the product separated magnetically into sponge iron for briquetting, an intermediate product, which is returned to the reducer, and a non-magnetic product, comprising the greater part of the gangue. For an ore containing 44% Fe the energy consumption per ton of iron was 7300 kw.-hrs., the coke consumption 312 kg., and the oil consumption 44·3 kg. The sponge iron produced contained 0·31% C, 0·16% Mn, 0·032% P, 0·013% S, 1·57% SiO_2, and 1·37% O, and the recovery was over 90%. The analyses of soft iron and mild steel obtained by melting the briquettes are given, together with details of their mechanical properties. In the Wiberg process the reduction is carried out in shaft furnaces, into which the gas, heated at 1100° in the regenerator, is passed; part of the gas is withdrawn from the middle of the shaft, cleaned from suspended dust, and passed through a coke layer in the regenerator, whilst the remainder passes up to the cooler parts of the shaft and effects a preliminary reduction of the freshly charged ore. The consumption of reducing agent and of electrical energy in this process is very similar to that of the Edwin process, but the rate of production of sponge iron is much lower. Methods involving reduction with solid carbon require a much simpler apparatus, and hence entail smaller heat losses, and consequently the costs of production are much lower. A. R. Powell.

Graphitising behaviour of iron carbide in pure iron carbon alloys in the critical range. H. P. Evans and A. Hayes (Trans. Amer. Soc. Steel Treat., 1927, **11**, 691—710).—Pure iron–carbon alloys containing 2·34% C were made by melting together Armco iron and pure Acheson graphite. When heated in a slightly oxidising mixture of carbon monoxide and dioxide at a pressure of 5 atm. at a temperature of 700—1080° graphitisation occurred, showing that iron carbide was metastable. The density fell during treatment from 7·80 to 7·66. The graphitisation at normal pressure was less than at 5 atm. T. H. Burnham.

The A3 stable transformation. H. A. Schwartz (Trans. Amer. Soc. Steel Treat., 1927, **11**, 767—780).—From observation of the stable A1 point of an iron–carbon–silicon alloy it was found that 1% of silicon raised it 17·6° compared with 16·6° for metastable alloys. By extrapolation the A3 stable point approaches 799° as a limit. Experiments with addition of nickel and other elements indicate the same temperature with sufficient accuracy. The A3 stable point is so close to that of A2 in pure iron that the β-phase is thought not to exist in the stable system. It is suggested that whilst austenite contains carbon in solution in the form of one atom of carbon in each molecule, boydenite contains

more than one and probably three atoms of carbon per molecule. T. H. Burnham.

Effect of stress on the decomposition of austenite. R. L. Dowdell and O. E. Harder (Trans. Amer. Soc. Steel Treat., 1927, **11**, 781—790).—Bars of alloy steels quenched to give an austenitic–martensitic structure were tempered at 100—200° under a bending stress less than their yield point. A permanent stress resulted but no difference of microstructure on the tension and compression sides. On upsetting specimens of austenitic structure at room temperature prominent slip lines were produced, but not the characteristic acicular martensitic structure. Tensile stress produced by forcing rings on a taper rod produced characteristic martensite. Austenitic specimens deformed in tension and subjected to —57° were transformed to martensite, whilst unstressed specimens were unchanged. Hammered austenitic specimens were transformed more readily on heating than unstressed ones. It is concluded that deformed austenite is rendered less stable on heating or on cooling. T. H. Burnham.

High-silicon structural steel. H. W. Gillett (U.S. Bur. Stand. Tech. Paper No. 331, 1926, **21**, 121—143).—A number of analyses of "Freund" steel showed a content of about 0·12% C, 0·5—0·8% Mn, and 0·8—1·2% Si. This steel is at present made in the Bosshardt furnace in Germany, but the physical properties (high yield-point and high ductility) are purely functions of the chemical composition, samples made in other furnaces giving similar results. The content of phosphorus, sulphur, and oxygen does not differ from that of other open-hearth steels. American high-strength structural steel on the contrary usually contains 0·3% C and about 1·0% Mn. The specimens of Freund steel (*d* 7·78) examined unetched showed much finely-divided sulphide, but few silicate inclusions. The combination of yield-point and ductility was better than that found in high-carbon steels of normal manganese content. The properties were, however, closely matched by reducing the carbon content and increasing manganese. Silicon in quantities above 0·5% is considered to be a true alloying element, and may be looked upon as equivalent to manganese, the choice between the two depending on economic considerations. C. Irwin.

Temperature measurements in liquid iron and steel. M. Wenzl and F. Morawe (Stahl. u. Eisen, 1927, **47**, 867—871).—The temperature of a bath of molten steel or iron in the reverberatory or blast furnace may be measured by means of a platinum–platinum-rhodium thermocouple enclosed in a thin quartz tube inside a "silite" tube; for temperatures up to 1,350° a nickel–iron couple may also be used. Temperatures taken with a Holborn–Kurlbaum optical pyrometer during casting are approximately 10° lower than those taken with a thermocouple, provided that the bright spots in the metal stream are focussed. The temperature of a stream of molten steel usually appears to be lower than that of the metal in the mould after removal of the slag layer, if the measurements are taken with an optical pyrometer; this is probably due to the presence of an oxide skin on the molten stream. A. R. Powell.

Macro- and microstructure of blowhole segregations in steel. A. Wimmer (Stahl u. Eisen, 1927, **47**, 781—785).—The recurrence and mode of formation of blowholes in steel and iron are discussed with reference to the composition and microstructure as revealed by various etching reagents. The results appear to indicate that sulphide and oxide segregations are the chief cause of blowholes as, in all cases, the sulphide inclusions are much more numerous around the blowholes than in the remainder of the metal. On the basis of observations on the distribution and arrangement of the various slag inclusions in steel and iron, a diagram of the ternary system, iron–ferrous oxide–ferrous sulphide, has been constructed in which the possibility of the formation of a ternary eutectic is suggested. A. R. Powell.

Magnetic analysis of high-speed steel. T. Spooner (Proc. Amer. Soc. Testing Materials, 1927, **26**, ii, 116—147).—The results of resistivity and various magnetic tests of high-speed steel are plotted against the quenching temperatures. Quenching temperatures between 1211° and 1296° gave similar microstructure and hardness, but different magnetic results. The most effective quench was obtained after 3—4 min. at the high temperature. The quenched bars were drawn in a salt bath at 538—621°, and the results of magnetic tests at once and after ageing are recorded.

Chemical Abstracts.

Treatment of metallic surfaces with aluminium. E. Neumann (Zentr. Hütten- u. Walzw., 1926, **30**, 554—556; Chem. Zentr., 1927, I., 945).—Treatment of the surface of iron and steel articles with aluminium renders them immune from scaling up to 1000°; nickel or nickel-chromium alloys may similarly be protected from oxygen and gases containing sulphur compounds up to the m.p. of the metal. The coating process involves heating the article at a high temperature in a mixture of aluminium turnings, alumina, and ammonium chloride in a closed container. A. R. Powell.

Analyses of copper-refining cell voltages. E. W. Rouse and P. K. Aubel (Amer. Electrochem. Soc., April, 1927, **51**. Advance copy. 9 pp.).—In the electrolysis of a copper-refining electrolyte containing 185·5 g. of sulphuric acid, 42 g. of copper, 14·8 g. of nickel, 3·8 g. of arsenic, and 0·5 g. of iron per litre with current densities of 1·6 and 1·9 amp./dm.^{2} at 57° using two copper electrodes 4 cm. apart, measurements have been made of the polarisation at anode and cathode, the voltage drop in the electrolyte, the total cell voltage, and the effect on these quantities of varying separately the temperature, the concentrations of sulphuric acid, copper, nickel, arsenic, and iron, and of adding small quantities of chloride (as hydrochloric acid) or glue to the electrolyte. The results are summarised by a series of graphs. The total cell voltage at given current density is mainly determined by the temperature and the concentrations of sulphuric acid and copper. Decreasing the copper content decreases the resistance, but increases the cathode polarisation, the latter effect becoming very considerable when the concentration falls below about 15 g./litre. Decreasing the nickel concentration reduces the voltage drop in the electrolyte, but causes an appreciable increase in cathode polarisation;

as a result of these opposing effects, the total cell voltage has a minimum value for about 13 g. of nickel per litre. Very small concentrations of glue raise the cathode polarisation considerably, so that addition of 10 mg./litre raises the total cell voltage by 60%. Added chloride acts as a cathodic depolariser. H. J. T. ELLINGHAM.

Addition agents in electro-deposition. G. FUSEYA and M. NAGANO (Amer. Electrochem. Soc., April, 1927, **51**. Advance copy. 15 pp. Cf. Fuseya and Murata; B., 1926, 950).—The effect of addition of glycine on the electrodeposition of copper from a *M*-copper sulphate solution has been investigated. Cathodic deposits from such solutions weigh more than the quantity of copper corresponding to Faraday's law, and assuming that the excess is due to glycine in the deposit, the concentration, c_s, in mols. of glycine per mol. of copper in the deposit can be determined. With neutral solutions at 17° and 0·04 amp./cm.^{2}, c_s increases from 0·015 to 0·05 when c, the concentration of glycine in the solution (mol./mol. of copper), is increased from 0·00125 to 0·10, c_s being greater than c until c exceeds 0·01. When c = 0·01, increasing the current density from 0·01 to 0·1 amp./cm.^{2} causes first a rapid and then a slower increase in c_s. Increasing the temperature of such a solution from 17° to 35° at 0·04 amp./cm.^{2} causes an almost linear fall in c_s from 0·0236 to 0·001. Acidifying this solution with sulphuric acid causes only a slight fall in c_s until the acid concentration exceeds about 0·001 *M*, when there is a rapid fall, and with *M*-sulphuric acid concentration the effect of the glycine on the weight of the deposit is entirely eliminated. Under all conditions the physical character of the deposit seemed to be determined solely by the magnitude of c_s; size of crystals diminishes with increase of c_s. In an attempt to relate the above phenomena to the concentration of complex ions in the solution, measurements were made of the *E.M.F.* of cells of the type, $Cu/M\text{-}CuSO_4/M.\ CuSO_4$ + glycine (concentration c)/Cu. The glycine lowers the copper-ion concentration, c_o, to an apparent value, c_o', such that $(c_o - c_o')/c_o = 1 \cdot 3\ c$, approximately. H. J. T. ELLINGHAM.

Reversed potentials in the corrosion of tin plate. C. L. MANTELL and W. G. KING, JUN. (Amer. Electrochem. Soc., April, 1927, **51**. Advance copy. 7 pp.).—In a study of the conditions for the corrosion of the tin plate of cans containing food products, measurements were made of the *E.M.F.* between iron and tin electrodes in freshly-opened cans of various food products (Mantell and Lincoln, Canadian Chem. Met., Feb., 1927). In foods preserved in saline solutions, the *E.M.F.* soon fell to zero, and then increased to a maximum in the reverse direction, the iron becoming nobler than the tin, apparently owing to the formation of a passivity film. This *E.M.F.* reversal did not occur with foods put up in sugar solutions. *E.M.Fs.* have now been measured between iron and tin electrodes in various simple solutions, and their variation followed during a period of 5 min. In strong electrolytes, the *E.M.F.* reverses rapidly, the iron being ennobled and thus protected against corrosion. In organic acids, such as formic, acetic, and citric, the *E.M.F.* falls rapidly at first, but a partial recovery occurs, any reversal of *E.M.F.* being temporary. In sucrose or carbamide solutions the small initial *E.M.F.* falls away to zero, and no reversal occurs unless a strong electrolyte is added to the solution. Solutions in which reversal of *E.M.F.* occurs are those in which perforation of the container by electrolytic action is rare. Foods put up in sugar solutions are most liable to give rise to perforation troubles. H. J. T. ELLINGHAM.

Age-hardening aluminium alloys; replacement of silicon with germanium. W. KROLL (Metall. u. Erz., 1926, **23**, 684—685; Chem. Zentr., 1927, I., 1061).—Germanium may replace silicon in alloys of the type of duralumin, aludur, and lautal, but its age-hardening action is not nearly so powerful. The mechanism of the action appears to be similar to that of the silicon alloys, but the ageing must be effected at 150°.
A. R. POWELL.

System germanium–aluminium. W. KROLL (Metall. u. Erz., 1926, **23**, 682–684; Chem. Zentr., 1927, I., 1061).—A thermal and microscopic examination of aluminium–germanium alloys with 0—60% Ge indicates the absence of compounds and solid solutions, and the presence in all alloys of a eutectic with 55% Ge, m.p. 423°. The presence of magnesium in the alloys leads to the formation of Mg_2Ge, which forms a solid solution with aluminium at high temperatures.
A. R. POWELL.

PATENTS.

Steel alloy. W. OERTEL, Assr. to GLOCKENSTAHLWERKE A.-G. VORM. R. LINDENBERG (U.S.P. 1,630,448, 31.5.27. Appl., 31.7.24. Conv., 9.1.22).—The alloy, which is highly resistant to chemical action and of great strength, consists of 8—25% Cr, 0·1—1·2% C, 0·2—6·0% Mo, 0·5—2·0% Ni, and remainder iron. The addition of the nickel renders the alloy more ductile when hot. F. G. CROSSE.

[Copper–iron] alloys. T. D. KELLY (E.P. 270,553, 22.9.26).—Alloys containing 10—90% Cu, 90—10% Fe, and up to 10% of nickel, chromium, or other metal commonly alloyed with iron are prepared by rapidly melting the charge of metal or, in certain cases, of metallic oxide in an electric furnace in the presence of non-oxidising fluxes, *e.g.*, cryolite, fluorspar, etc.
C. A. KING.

Solder for fixing in position lead-bearing metal containing alkali or alkaline-earth metals. A. WERNER (G.P. 438,392, 4.12.24).—The solder comprises 2 pts. of lead, 2 pts. of tin, and 0·2% P. The latter is preferably added as phosphor–tin. A. R. POWELL.

Separation of zinciferous pyrites into its constituents. H. SCHUMACHER (G.P. 437,891, 29.2.24).—Pyritic iron ore containing zinc is roasted and the product mixed with an oxidised iron ore or with the residue from leaching roasted cupriferous pyrites with sulphuric acid. The mixture is sintered by roasting with a small proportion of the original pyrites, and the product is smelted in a blast furnace to obtain pig iron and zinc oxide fumes. A. R. POWELL.

Recovery of zinc oxide from furnace gases. Manufacture of zinc oxide. J. F. CREGAN, Assr. to AMERICAN SMELTING AND REFINING CO. (U.S.P. 1,628,952—3, 17.5.27. Appl., [A], 12.8.22; [B], 25.10.24).—(A) The

zinc fume formed in a reverberatory furnace is subjected to a reducing atmosphere at a high temperature by means of a reducing gas; the resulting metallic fume is subsequently oxidised. (B) The vapour distilled from the smelting of material containing zinc is passed into a settling chamber to discharge impurities, after which it is removed and mixed with air and converted to the oxide within a combustion chamber.

H. Royal-Dawson.

Platinum metal alloys for tipping the nibs of fountain pens. W. C. Heraeus G.m.b.H., Assees. of E. Haagn (G.P. 437,173, 17.9.25).—The alloys consist of 40—60% Ru, 35—50% Os, and 5—15% Pt.

A. R. Powell.

Concentration [flotation] of ores. C. H. Keller, Assr. to Minerals Separation North American Corp. (U.S.P. 1,628,151, 10.5.27. Appl., 24.11.25).—Potassium ferricyanide is added to sodium xanthate solutions used in ore flotation. T. S. Wheeler.

Ore flotation process. J. Herman, A. W. Allen, and H. R. Newitt (U.S.P. 1,628,046, 10.5.27. Appl., 25.11.25).—Ore pulp containing oxygen compounds of metals is subjected to the usual flotation process, but in place of air, a reducing gas, *e.g.*, coal-gas, is employed to provide an ascending stream of bubbles.

T. S. Wheeler.

De-watering flotation slimes. F. Krupp Grusonwerk A.-G. (G.P. 435,890, 29.3.23).—Flotation slimes are filtered on a bed of coarse-grained material which is given a shaking motion in order to force the coarse particles into the froth and break the bubbles.

A. R. Powell.

Concentration of minerals from ores by the use of sulphones as flotation reagents. F. Laist and F. F. Frick (U.S.P. 1,629,080, 17.5.27. Appl., 13.4.26). —Sulphones, *e.g.*, diphenylsulphone, are of value as froth-flotation reagents. T. S. Wheeler.

Manufacture of plated articles. E. G. Bek (E.P. 244,487, 11.12.25. Conv., 11.12.24).—Copper, bronze, brass, or nickel-silver alloys are plated with gold or silver from any of the well-known plating baths, the deposit is washed and coated with a strong solution of boric acid and/or borax, and the article is then heated at such a temperature above 450° and for such a time (2—5 min.) that the desired colour is imparted to the surface of the article. If desirable, in order to obtain a modified colour, the article may be given a preliminary coating, by electrodeposition of one or more layers of a metal, such as copper or nickel, which will diffuse into the gold during the heating operation. A. R. Powell.

Coating metal surfaces (U.S.P. 1,629,072).—See VIII.

XI.—ELECTROTECHNICS.

Law of alternating-current electrolysis and the electrolytic capacity of metallic electrodes. J. W. Shipley and C. F. Goodeve (Amer. Electrochem. Soc., April, 1927, **51**, Advance copy. 25 pp.).—Previous work (Eng. J. Canada, 1927, **10**, 3) showed that, in *A.C.* electrolysis using given electrodes under given conditions, there is a certain critical current density which must be exceeded before gas evolution begins, but at higher current densities the excess of current above that corresponding to the critical density generates electrolytic gas in quantity required by Faraday's laws. Investigations have now been made at 30° in $0 \cdot 5N$-sodium hydroxide using 60-cycle *A.C.* and electrodes of various metals held between rubber insulators so as to avoid exposure of points and edges at which the current density might vary. The above conclusions were generally supported, except that, at current densities just above the critical, deviations from the quantitative law were found with platinum and silver and especially with nickel. The observed critical current densities were: Pt, 0·7; Ag, 1·6; Cu, 1·9; Fe, 3·8; steel, 4·5; Ni, 4·6 amp./cm.^{2} For aluminium the value, if existing, must be less than 0·01 amp./cm.^{2} In many cases it was found possible to exceed the critical density without gas evolution if the current were very gradually raised, but when gas evolution did begin its rate rose sharply to the normal value for that current density. In such cases of delayed gas evolution, oxide films were observed, especially with copper, but they disappeared when gas was evolved. With an oxidised copper electrode the apparent critical current density was 10 amp./cm.^{2} With corroded steel there was a true critical current density of 8 amp./cm.^{2} Assuming that the existence of a critical current density is due to an "electrolytic capacity" of the electrodes for storing up the products of electrolysis, values of this capacity are calculated for various metals, and the figures so obtained agree with those furnished by direct measurement of the maximum quantity of electricity obtainable by completely discharging an electrode immediately after breaking the electrolysis circuit. The electrolytic capacity represents the average of the quantities of electricity per cm.^{2} which must pass after the theoretical decomposition voltage is reached before gases are liberated from each of two electrodes; the value for iron was about 0·014 coulomb/cm.^{2} Preliminary work on the effect of frequency and temperature on electrolytic capacity is recorded. The relation between electrolytic capacity and "polarisation capacity" (Kohlrausch) is discussed, and it is concluded that the latter is negligible compared with the former in these experiments. The relation of the back *E.M.F.* to the quantity of electricity passed was determined for platinum electrodes and related to the voltage wave of the *A.C.* The curve of resultant voltage is distorted and out of phase with the current wave, the current leading the voltage. The experiments were undertaken to find the conditions under which production of explosive gases in electric boilers can be avoided. Current densities used in such boilers are well below the critical value for steel, and explosion risks only arise from concentration of current on small areas. In the previous paper (*l.c.*) designs of electrode chambers leading to conditions of uniform current density were given. H. J. T. Ellingham.

Miguet electrode and the Miguet furnace. M. Arrouet (Amer. Electrochem. Soc., April, 1927. Advance copy. 4 pp.).—For the manufacture of ferro-alloys, calcium carbide, etc. three-phase furnaces have been preferred, but, when properly designed, large single-phase furnaces are practical and efficient. The design of the Miguet furnace enables the current to be led to a single

electrode through a number of separate circuits arranged symmetrically round the furnace. The current in each circuit being relatively small, though the total may reach 240,000 amp. The bus-bar interlacing is brought very close to the furnace so that the loop is exceptionally small and the power factor higher than has hitherto been attained with high-power single-phase furnaces. The Miguet continuous electrode is built up of previously baked carbon segments assembled above the furnace, and is fed into the furnace by releasing the pressure on the bronze supporting plates which also conduct the current to the electrode. A reducing agent such as coke is found to give maximum efficiency if the furnace is fed with alternate layers containing excess of carbon and excess of material to be reduced. The current is thus spread over a larger reaction zone, and heating is entirely by resistance. The bottom of the electrode is kept about 8 in. above the bath, so that the current passes only at the edge of the circular reduction zone, the area under the electrode being available for special refining reactions. A 5000 kw. unit operated in France during 1926 required only 3100 kw.-hrs. per metric ton of calcium carbide produced, and a saving of raw materials as compared with three-phase furnaces is claimed. H. J. T. Ellingham.

Chemical method for the standardisation of ultra-violet light. J. E. Moss and A. W. Knapp (Brit. J. Actinotherapy, May, 1927. Reprint. 4 pp.).—The standard Uroxameter apparatus cannot be used with vertical carbon arcs because it cannot be placed close enough to the arc. A modified apparatus is described using a circular flat-bottomed dish 15 cm. in diameter and 1·8 cm. high. The ratio of uranium salt to oxalic acid in the solution was increased 20 times, and contained 25 c.c. of a 2% aqueous solution of uranium acetate, 10 c.c. of a 2% aqueous solution of oxalic acid, and 15 c.c. of water. The depth of the solution, which must be constant in all tests, was 0·23 cm., and the dish was placed 12 in. from the arc in a direction 45° to the vertical. After a 15 min. exposure the excess of oxalic acid was titrated with permanganate. Comparative results on different types of arc are given.

C. J. Smithells.

Progress of electrometric control methods in industry. H. C. Parker (Ind. Eng. Chem., 1927, **19**, 660—667).

Corrosion of tin plate. Mantell and King.—See X.

Copper-refining cell voltages. Rouse and Aubel.—See X.

Addition agents in electrodeposition. Fuseya and Nagano.—See X.

Patents.

Electric incandescence lamp [getter]. W. C. Sproesser, Assr. to Westinghouse Lamp Co. (U.S.P. 1,624,077, 12.4.27. Appl., 3.12.21).—A getter mixture containing silica (40%), cryolite (50%), and phosphorus (10%) is applied to the filament before flashing.

T. S. Wheeler.

Electric incandescence lamp. D. MacRae, Assr. to Westinghouse Lamp Co. (U.S.P. 1,624,109, 12.4.27. Appl., 2.12.21).—A method of eliminating flashing of the filament in the manufacture of incandescence lamps, comprises exhausting the lamp sufficiently to render the residual gas non-conducting at the working voltage, and employing as a getter a substance, *e.g.*, cryolite, which on burning is completely vaporised without evolution of sufficient permanent gas to render conducting the atmosphere in the lamp. T. S. Wheeler.

Getter for controlling crystal growth. Introduction of clean-up agents into evacuated vessels. Application of getters to electric incandescence lamps and the like. W. Sproesser, Assr. to Westinghouse Lamp Co. (U.S.P. 1,626,637—9, 3.5.27. Appl., [a] 6.12.22; [b] 5.5.23; [c] 21.8.23).—(a) A getter for application to thoriated tungsten filaments comprises phosphorus (13 pts.), cryolite (75 pts.), and thorium oxide (25 pts.). (b) Phosphorus pentoxide (1 pt.) is fused with cryolite (1 pt.), and the product, which does not absorb moisture, is mixed with an equal weight of powdered aluminium, and employed as a getter. On flashing, free phosphorus is liberated. (c) The getter is placed on an auxiliary filament of platinum, which is temporarily sealed in the lamp and flashed. The auxiliary filament is then replaced by the actual filament. T. S. Wheeler.

Evacuation of [electric lamp] bulbs and the like. W. C. Sproesser, Assr. to Westinghouse Lamp Co. (U.S.P. 1,626,640, 3.5.27. Appl., 7.9.23).—Electric lamp bulbs are evacuated by passing through them at about 400° a current of the vapour of aluminium chloride, or like substance possessing an inappreciable vapour pressure at ordinary temperatures. T. S. Wheeler.

Optical pyrometer. Siemens Brothers & Co., Ltd., and L. G. Salmon (E.P. 270,901, 12.5.26).—In optical pyrometers of the disappearing filament type, a circular scale extending over a comparatively large arc, *e.g.*, one extending over 240°, is employed in the indicating instrument. Preferably the portion of the scale corresponding to the non-luminous condition of the filament is masked. J. S. G. Thomas.

Positive electrode for electric batteries. R. Oppenheim, Assr. to Soc. Anon. le Carbone (U.S.P. 1,631,642, 7.6.27. Appl., 3.1.25. Conv., 19.11.24).—See E.P. 243,300; B., 1926, 446.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

"Crystallising-out" of solutions of driers. Anon. (Farben-Ztg., 1927, **32**, 2020—2021).—The "throwing out" of driers from solutions in light petroleum, benzene, and turpentine, etc. either by precipitation (lead driers in general) or by true crystallisation (cobalt linoleate etc.) is described in detail. The change is shown to proceed from the surface in all cases, and is attributed to the production of insoluble substances by atmospheric action. The influence of composition and of the method of preparation of the driers (*i.e.*, fusion or precipitation), both of which have been advanced as causes of this "crystallisation," are shown to affect concentration of solution only, and to afford no true explanation of the phenomena in question.

S. S. Woolf.

Vanadium compounds as driers. F. HEBLER (Farben-Ztg., 1927, **32**, 2077—2078).—Polemical with Swehten (B., 1927, 259) and with Rhodes and Chen (B., 1922, 335 A). Vanadium driers accelerate the rate of oxygen-absorption of linseed oil films, but retard their actual drying. Boiled oil films containing 0·1% and 0·2% of vanadium remained tacky for more than 1 week, whilst the original raw linseed oil dried in 5 days. The wrinkling of films reported by Swehten is also not confirmed, and his experimental conditions are queries. The field of application of vanadium as a commercial drier is considered to be problematical.

S. S. WOOLF.

Nitrocellulose for use in lacquers. E. VON MÜHLENDAHL and H. SCHULZ (Farben-Ztg., 1927, **32**, 2021—2022).—Stability, solubility, and viscosity determinations to be carried out on nitrocellulose are described. S. S. WOOLF.

Investigation of resins with *X*-rays. S. VON NÁRAY-SZABÓ (Biochem. Z., 1927, **185**, 86—87).—The behaviour of various resins towards *X*-rays indicates that they exist in all grades of conditions from the amorphous to the crystalline. Crystalline resins, particularly of the benzoin type, show well-marked differences corresponding to their different chemical compositions.

C. R. HARINGTON.

PATENTS.

Manufacture of condensation products of crotonaldehyde. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 270,433, 15.2.26).—The resinous condensation product of crotonaldehyde and an aromatic amine in the absence of an acid condensing agent is claimed. A suitable solvent or diluent may be present, *e.g.*, alcohol, benzene, acetone. S. S. WOOLF.

Production of coloured [transparent] polymerised styrene and its homologues. I. OSTROMISLENSKI, Assr. to NAUGATUCK CHEMICAL Co. (U.S.P. 1,627,195, 3.5.27. Appl., 29.1.25. Cf. E.P. 233,649; B., 1926, 451).—Colouring materials soluble in polymerised and unpolymerised styrene, stable about 175° and unaffected by formaldehyde, which is formed in the polymerisation of styrene, are dissolved in styrene, which is then heated at 140—175° to induce polymerisation. *E.g.*, anthracene (0·5%) in styrene gives a violet fluorescence to the polymerised material, without affecting its transparency. T. S. WHEELER.

Manufacture of resin-like substances from cracked hydrocarbon products. J. C. MORRELL and G. EGLOFF, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,627,054, 3.5.27. Appl., 27.4.23).—The coloured resinous substances which are removed by adsorbents from cracked petroleum distillates are treated with air at a slightly elevated temperature, and with polymerising agents, *e.g.*, the salts of heavy metals. Hard resins of value in the manufacture of varnishes are obtained. T. S. WHEELER.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Ultramicroscopic studies on theory of vulcanisation by H. Dannenberg. New effect of light in the system rubber–sulphur by H. Pohle. F. KIRCHHOF (Kautschuk, 1927, 184).—The formation of nuclei of colloidal sulphur in solutions of sulphur in rubber under the influence of light of short wave-length is attributed to the occurrence of ionised atoms or positive ions which arise from the action of the radiation on the neutral sulphur atoms. The effect is regarded as analogous to that of the ionisation of gases by α- or β-rays in moist air with formation of mist. An analogous phenomenon is observable in a solution of rubber and phosphorus in benzene. The marked increase in absorption of light by rubber on vulcanisation is ascribed to the formation of the colloidal sulphur.

D. F. TWISS.

Measurement of abrasion-resistance of rubber. I. WILLIAMS (Ind. Eng. Chem., 1927, **19**, 674—677).—The standardised conditions of surface contact, load, and degree of slippage in most abrasion-testing machines do not ensure the expenditure of a comparable amount of work with different samples. A machine is described enabling the measurement of the power consumed, the results being expressed in volume loss per unit of work done. The machine comprises a rotating disc with an abrasive surface against which two test blocks of the rubber are pressed with known force; measurement is also made of the force necessary to keep the blocks stationary. Results are quoted for tensile and abrasion tests on five mixtures of comparable composition, but containing new and "reclaimed" rubber in the proportions, 100 : 0, 75 : 25, 50 : 50, 25 : 75, and 0 : 100, the relative values for abrasion resistance at an equal rate of work being 1·0, 0·77, 0·55, 0·37, and 0·21 respectively. D. F. TWISS.

Tables of comparison for steam pressures and temperatures in vulcanisation. W. ESCH (Kautschuk, 1927, 156—158).—Steam-pressure gauges calibrated to give a reading of 1 kg./cm. at 100° show a discrepancy of 0·5 lb./sq. in. relative to the value of 14·7 lb./sq. in. for the pressure of 1 atm. Tables are given for the inter-conversion of steam pressures and the corresponding temperatures. A table is also given showing equivalent periods of vulcanisation at different temperatures, its value being illustrated by the vulcanisation behaviour of several mixtures. D. F. TWISS.

Physiological considerations on the latex-flow of rubber plants. A. ZIMMERMANN (Kautschuk, 1927, 95—98, 118—121, 147—149).—A critical survey of the knowledge of the character and features of latex-flow under various conditions. D. F. TWISS.

Lamp-black in rubber. A. VAN ROSSEM and H. VAN DER MEYDEN (Kautschuk, 1927, 166—174).—A comparative examination of several grades of lamp-black and one of carbon black prepared from natural gas, as to their chemical composition, sp. gr., relative bulkiness, behaviour on sieving, appearance under the microscope, and their effect on the strength, extensibility, energy of resilience, ageing, and abrasion-resistance of vulcanised rubber in which they have been incorporated.

D. F. TWISS.

PATENTS.

Regeneration of rubber and particularly that contained in the skeletons of disused tyres. C. DANIER (E.P. 269,127, 22.9.26. Conv., 8.4.26).—

Waste material with a small content of desirably pure rubber and a larger proportion of canvas (*e.g.*, pieces of pneumatic tyre) is impregnated with the liquid obtained by the destructive distillation of otherwise worthless rubber scrap (*e.g.*, beads of disused motor covers, old rubber tubing, etc.). The impregnated material is then immersed (for from 15 to 20 days) in concentrated hydrochloric acid at the ordinary temperature, which causes destruction of the canvas and also effects the polymerisation of the absorbed distillate into synthetic rubber. After being washed and crêped the product can be used instead of new rubber for all purposes except for making rubber solution. D. F. TWISS.

Preservation of goods of rubber or like substances. B. D. PORRITT, T. R. DAWSON, and RESEARCH ASSOC. OF BRIT. RUBBER & TYRE MANUFRS. (E.P. 269,745, 25.5.26).—In the manufacture of goods of rubber there are added small proportions of substances which protect the rubber simultaneously against the effects of oxidation and of actinic light. The former effect is produced by antioxidants such as polyphenols and aminophenols, whilst the latter is produced by dyestuffs capable of absorbing ultra-violet light. D. F. TWISS.

Applying liquids [by means of porous rubber]. H. BECKMANN (E.P. 270,374, 27.1.26).—Rubber, or even vulcanite with microscopically small pores, is used for making rollers, *e.g.*, for use in printing machines, or pads or wicks to apply oil to bearings. Such rubber with capillary pores can be produced by coagulating a mixture of latex and sulphur with magnesium sulphate, and vulcanising the resultant firm jelly. D. F. TWISS.

XV.—LEATHER; GLUE.

[Polarisation microscope for controlling] tanning and the penetration of the tannin. J. JOUANOVITS (Collegium, 1927, 226—236).—Pieces of limed South American hides were washed, delimed with formic acid, and used for the experiments. Collagen fibres 1—2 cm. long were isolated. They consisted of bundles of fine fibrils 5—8μ in diameter with blunt bulbous ends, and showed the property of double refraction. A smaller number of other coarser fibres were found, thickness 8—15μ, which were like corkscrew bands, and closely resembled cotton fibres in appearance, and were strongly refractive. These are possibly the so-called "elastic fibres." Heat gelatinises the collagen fibres mounted in water, the change occurring suddenly with the fibre bundle turning on its own axis, contracting towards the middle, and finally forming a short swollen cylinder, the power of double refraction being lost. The elastic bands are not affected by the heat. Acids and alkalis are both similar to gelatinisation in their effects. During tannage the fibre bundles become much darker in colour as seen through the microscope, and then later the polarised colour of the fibre bundles becomes weaker and is finally lost, and an even dark brown colour remains. With pyrogallol tannins, the first phase of the tannage causes a reversal of the double refraction, but with the progress of the tannage this diminishes and finally fails leaving an even dark brown colour. The physical properties of the elastic fibres are not affected by tannage. The polarisation microscope can be used to follow the progress of the tannage from liquor to liquor, and to determine whether the leather is sufficiently tanned. Then sections of insufficiently tanned leather, mounted in water or glycerin–gelatin medium, show the brightly illuminated interference colours of the raw hide fibre when examined under the instrument. Complete penetration of the tannin is revealed by a few isolated, intense green, fibre bundles in the positive position with sole leather. D. WOODROFFE.

Determination of moisture in leather. [American Leather Chemists' Association's Committee Report.] F. P. VEITCH and T. D. JARRELL (J. Amer. Leather Chem. Assoc., 1927, **22**, 265—274).—The moisture was determined in samples of chrome- and vegetable-tanned, stuffed and unstuffed leathers respectively, by the official method and the toluene distillation method (B., 1926, 926). It was found that there was a small continued loss in weight with any kind of leather upon repeated drying by the official method. The results by the latter method were affected by the relative humidity of the air unless a current of dry air was passed through the drying bottle during the drying. Variations in the temperature of the drying ovens caused variations in the results. The highest moisture figures were obtained with the vacuum oven at 105°. The results obtained by the toluene distillation method and the official method did not agree well, difference in types of ovens, in temperature, and humidity within the ovens causing large differences between the results of different analysts. Greater concordance was evidenced by the toluene distillation method than by the official method. D. WOODROFFE.

Action of trypsin on unlimed calfskin. H. B. MERRILL and J. W. FLEMING (J. Amer. Leather Chem. Assoc., 1927, **22**, 274—278).—Samples of limed and unlimed calfskin from the same pelt were prepared, treated with a 0·01% solution of commercial trypsin in a buffer solution of p_H 8·0 at 40°, and the nitrogen content was determined after 24 hr. periods up to 8 days. Both the limed and unlimed skin were hydrolysed more rapidly during the first two or three days, the rate of hydrolysis gradually falling off to a constant value. Limed calfskin contained a greater amount of material easily digested by trypsin, but after its removal the rate of hydrolysis of the collagen was the same as for the unlimed skin. The rate of digestion of collagen by trypsin is independent of whether or not it has previously been limed. D. WOODROFFE.

Badan. R. W. FREY and L. R. LEINBACH (J. Amer. Leather Chem. Assoc., 1927, **22**, 243—244).—A sample of badan root (*Saxifraga crassifolia*) obtained from Russia analysed as follows, on a moisture-free basis:—Tans, 26·8%; non-tannins, 23·7%; insolubles, 2·4%; sugars before hydrolysis, 6·6%; after hydrolysis, 13·8%. The presence of a mixture of pyrogallol- and pyrocatechol-tannins was indicated. A cow-grain skiver tanned in the aqueous extract had a fawn colour. D. WOODROFFE.

Insoluble constituents of myrobalan extract. P. CHAMBARD (Le Cuir Technique, 1926, **18**, 372—373; Chem. Zentr., 1926, II., 3132).—Insoluble residues

obtained by centrifuging the extracts contain microscopic crystals soluble in alcohol and in alkalis, but precipitated from solution in the latter by acidification. The crystals, which in neutral solution give a black coloration with iron salts and a precipitate with gelatin, are probably a crystalline form of the active tanning agent in the extract, resembling chebulinic acid.

L. A. Coles.

XVI.—AGRICULTURE.

Plant analysis as a means for determining the content of nutrients in soils. A. Jacob (Ernährung d. Pflanze, 1926, **22**, 300—302; Chem. Zentr., 1927, I., 792).—A discussion of the method proposed by Hoffer and Trost (Purdue Univ. Agric. Exp. Stat., Bull. 298) for detecting a shortage of potassium in soil by determining the extent to which accumulation of iron has taken place in the tissues of the stem of maize plants grown on the soil in question. The relative iron content is indicated by the depth of colour obtained on placing a few drops of a 10% aqueous solution of potassium sulphocyanide and dilute hydrochloric acid on the cut surface of the stem. If the majority of the plants show a high content of iron, the soil lacks potassium. Application of lime to the soil increases the iron content of the plants; kainit diminishes it, even if lime also is given.

C. T. Gimingham.

Manuring trials with potassium nitrate. F. Frowein (Chem.-Ztg., 1927, **51**, 341—343).—Field trials with potatoes indicated that potassium nitrate was as efficient as a mixture of sodium nitrate and potassium sulphate, both as regards total yield and starch content of tubers. Slightly greater yields of leaves of tobacco resulted from the use of potassium nitrate in comparison with the mixed fertiliser. Inferior yields were obtained with oats when potassium nitrate was used. Laboratory experiments with the Neubauer method showed that the intake of phosphorus by the plants was greater when potassium nitrate was applied than when mixtures of potash salts and the usual nitrogenous fertilisers were used.

A. G. Pollard.

Analysis of calcium nitrate. J. M. McCandless and J. Q. Burton (J. Assoc. Off. Agric. Chem., 1927, **10**, 216—219).—The analysis of synthetic calcium nitrate presents difficulty owing to the extreme hygroscopicity of the salt. It is recommended that lumps should be crushed and, without sieving, placed in stoppered bottle. 15 g. weighed rapidly in tightly-covered dish are at once brought into solution and diluted to 1 litre, of which 25 c.c. are treated with 0·4 g. of sodium carbonate, and evaporated to dryness to remove traces of ammonium nitrate, the residue being then treated with salicylic–sulphuric acid mixture and analysed by the Kjeldahl–Gunning method. Analysis of the salt by the reduced iron method gave low results, probably owing to evolution of nitric oxide with the hydrogen. The determination of moisture presents difficulty, since drying at 130° removes part of the water of crystallisation. Distillation with toluene (cf. Bidwell and Sterling; B., 1925, 268) and with xylene gave inconsistent results. The following method is recommended: Mix 10 c.c. of calcium nitrate solution (10 g. in 250 c.c. of water) with 10 c.c. of sodium carbonate solution (250 c.c. contain sufficient sodium carbonate to combine with all the calcium nitrate, factor 0·646), evaporate the mixture to dryness, dry at 130° for 3 hrs., cool, and weigh. The difference between the combined weights of salts taken and the weight of the dried residue is the weight of water in the calcium nitrate, allowance being made for the ammonium nitrate driven off.

H. J. Dowden.

Suggested modification of the official method for potash [determination] in mixed fertilisers. L. D. Haigh (J. Assoc. Off. Agric. Chem., 1927, **10**, 220—222).—The presence of phosphoric acid and soluble phosphates in fertilisers leads to a low assay in the determination of potash by the official method. Removal of phosphates by magnesium chloride may give a high assay (cf. Bible; B., 1925, 606). They can, however, be satisfactorily removed by heating the solution with calcium carbonate. Heat at the b.p. for 2—3 min. 2·5 g. of the fertiliser, 2 g. of calcium carbonate, and 75 c.c. of water. Evaporate down to 25 c.c., filter, wash, and to the filtrate add 1—2 c.c. of strong ammonia solution and sufficient ammonium oxalate solution to precipitate all the calcium. Cool, dilute to 250 c.c., and, after filtering, determine potash in the filtrate by the usual Lindo–Gladding method. H. J. Dowden.

Determination of the replaceable bases of soils, in either presence or absence of alkali salts. P. S. Burgess and J. F. Breazeale (Arizona Agric. Exp. Sta. Tech. Bull., 1926, 9, 187—203).—The methods are applicable to acid, neutral, or calcareous soils. 0·1*N*-Barium chloride solution is employed as the replacing agent, the barium being removed as chromate in the determination of calcium and magnesium, or as sulphate in that of sodium and potassium. A proposed method for the determination of replaceable bases in black alkali soils involves the determination of calcium only. Chemical Abstracts.

Reaction between calcium sulphate and sodium carbonate, and its relation to the reclamation of black alkali lands. J. F. Breazeale and P. S. Burgess (Arizona Agric. Exp. Sta. Tech. Bull., 1926, **6**, 125—129).—Soils containing black alkali contain no free carbon dioxide. When gypsum is applied as a corrective, the amount of black alkali cannot be reduced below about 25 pts. per million; the soluble salts should then be leached out. Application of organic matter and aeration permit the aerobic soil bacteria to produce carbon dioxide necessary to convert the small remaining quantity of sodium carbonate into the innocuous sodium hydrogen carbonate. Chemical Abstracts.

XVII.—SUGARS; STARCHES; GUMS.

Electrical conductivity of solutions of granulated sugars. [Determination of ash electrically.] A. R. Nees (Ind. Eng. Chem., 1927, **19**, 225—226).—The apparatus used consisted of a 1 : 1 transformer (utilising current from a 100-volt, 60-cycle lighting circuit), an alternating-current galvanometer, a dial-type Wheatstone bridge, and a conductivity cell, it being possible for one operator with assistant to make 150 conductivity determinations in 8 hrs. Determinations were carried out at 25°, using solutions containing 25 g. of sugar per 100 c.c.,

the results being expressed in terms of the sp. conductance (as multiples of 10^{-5}), this divided by the factor 231·5 in the case of beet sugars produced in Colorado, Nebraska, and Montana giving the % of ash. Variation of the conductance with the concentration of the sugar solution is very slight, being near the maximum at a concentration of 25 g. per 100 c.c. J. P. OGILVIE.

Use of the isoelectric point as guide to the neutralisation of converter liquor in starch-glucose manufacture. H. S. PAINE and M. S. BADOLLET (Facts about Sugar, 1926, **21**, No. 51).—Ultramicroscopic cataphoresis measurements were made of a number of samples of "converter liquors," and it was found that, on addition of sodium carbonate for neutralisation, maximum flocculation occurred in all cases at a definite p_H value (the isoelectric point), but that this value varied with different samples, and was apparently dependent on a number of factors, such as character of the raw material, conditions of hydrolysis, etc.

J. P. OGILVIE.

Determination of reducing sugars volumetrically. M. VAN DE KREKE (Archief Suikerind. Nederl.-Ind., 1926, 411—419; Int. Sugar J., 1927, **29**, 110).—Schoorl's iodine method is modified for application to cane molasses as follows: 50 c.c. of Fehling's solution are mixed with 50 c.c. of the solution of the sample, heated to b.p. in 3 min., and ebullition is continued for 2 min. longer. After cooling, 25 c.c. of 20% potassium iodide solution and 35 c.c. of sulphuric acid (1 : 5 pts.) are added, the iodine thus liberated being titrated with 0·1*N*-sodium thiosulphate, using starch indicator. A "blank," using water in place of sugar solution, is carried out, the figure found being deducted from that actually obtained. J. P. OGILVIE.

Bone black [animal charcoal]. H. I. KNOWLES (Ind. Eng. Chem., 1927, **19**, 222—225).—Shank and knuckle bones were carbonised, and the following percentage results found for the chars respectively: carbon, 7·26, 16·37; insoluble in hydrochloric acid, 0·12, 0·96; calcium carbonate, 9·49, 7·48; apparent sp. gr., 0·662, 0·502; colour removed (from a solution containing 1000 colour units), 96·9, 98·4; ash removed, 64·1, 60·0%. Carbon content is not always a criterion of char value, there being a difference in the quality of the carbon. There appears to be an optimum carbon content, depending on the available bone surface area, and for shank char this seems to be about 10%. Ash removal by char is selective, and, like colour removal, is an adsorptive phenomenon, from which it follows that tests for measuring colour and ash must take into consideration the concentration of these constituents. The Brix and p_H values are also important. A hardness test is described in which the char was subjected to the impact and abrasion of steel balls in a small cast-iron ball mill, the increase in "fines" passing both 24- and 50-mesh being measured. J. P. OGILVIE.

Decomposition of the invert sugar by lime [in the defecation of beet raw juices]. V. ČTYROKÝ (Z. Zuckerind. Czechoslov., 1927, **51**, 230—236).—In the decomposition of invert sugar by lime, the rate of decomposition depends firstly on the temperature, and secondly on the amount of lime. Under the conditions of practice almost the total invert sugar is destroyed during the time of contact of the juice with the milk of lime in defecation and saturation. This time practically suffices for the complete decomposition of amounts of invert sugar as high as 1%. J. P. OGILVIE.

XIX.—FOODS.

Rate of drying of wheat flour, starch, and gluten. E. A. FISHER (Cereal Chem., 1927, **4**, 184—206).—The rates of drying of different varieties of flour, starch, and crude powdered gluten were studied by exposing them over sulphuric acid at a constant temperature in an apparatus which allowed of their being weighed at intervals without removing them from the drying vessel. The curves obtained by plotting the rates of drying against moisture content are all of the same type. The rate of drying is a linear function of the moisture content down to about 6% of the latter. Between 6% and 2½% the curve is again linear, but different in direction, and below 2½% the rate of drying becomes extremely slow, the curve bending round to the origin. There is some evidence to suggest that the intercepts on the moisture axis cut off by the extension of the two straight-line portions of the curve are characteristic of each flour and connected with the amount of gluten present. The rate of drying is probably controlled largely by convection currents set up by differences of temperature between the drying material, the acid, and the parts of the apparatus in contact with the outside air. F. R. ENNOS.

Use of Ostwald viscosimeters for flour suspensions. H. J. DENHAM, G. W. S. BLAIR, and G. WATTS (Cereal Chem., 1927, 4, 206—220).—The Ostwald type of viscosimeter has been found satisfactory for measurements on flour suspensions of concentrations up to 30%, provided the instrument is calibrated with solutions of known viscosity and the measurements are expressed in absolute units. Varying the dimensions of the capillary within wide limits does not affect the accuracy of the results, provided the flow is laminar and the critical velocity is not exceeded. Viscosity "fall-back," or the tendency of the viscosity of flour suspensions to decrease on keeping, is shown to be a colloidal phenomenon, and is probably due to syneresis occurring in the suspensoid. From a study of the change of viscosity of flour suspensions with temperature, an empirical equation relating temperature coefficients with concentration has been put forward. F. R. ENNOS.

Vitamin-C content of fresh and canned pear. V. C. CRAVEN and M. M. KRAMER (J. Agric. Res., 1927, **34**, 385—392).—The minimum dose of raw pear required to protect guinea-pigs from scurvy is 10—15 g. per day. The antiscorbutic factor in pear is destroyed by the "open-kettle" method of home canning, involving boiling the fruit in water for 15 min., and is partly destroyed by the "cold-pack" method, in which the jars containing the fruit are kept in boiling water for 20 min. Storage of fresh pears seemed to make no difference in the content of vitamin-*C*.

C. T. GIMINGHAM.

Corrosion of tin plate. MANTELL and KING.—See X.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPT. 2, 1927.

I.—GENERAL; PLANT; MACHINERY.

PATENTS.

Ovens for baking, drying, and other heating operations. T. & T. VICARS, LTD., and E. M. CROSLAND (E.P. 272,261, 1.2.26).—The goods are conveyed through the oven in tiers (*e.g.*, on trays) which alternate with heat-radiating elements so that the goods are heated both above and below. The radiators are heated internally by burners which direct the heat either downwards or upwards as desired.

B. M. VENABLES.

Apparatus for drying material in bulk, or in stacks. C. TINKER (E.P. 272,316 and 272,319, [A], 6.4.26, [B], 15.4.26).—Hot air is supplied through a duct passing underneath and up through a drying floor, and the bulk material is stacked around the air outlet. In (A) a casing is erected so designed as to admit the hot air at different levels to the material. In (B) a liftable core is used to form an air shaft through the material as it is built up. B. M. VENABLES.

Utilisation of heat in drying drums. C. LUDWIG (E.P. 267,543, 11.3.27. Conv., 11.3.26).—In a rotary dryer which is provided with spiral air outlets at the dry end, the internal distributors for the material are continued right across the air outlet zone and the material is allowed to accumulate in a heap preparatory to discharge in a zone between air outlets and a baffle or end wall. B. M. VENABLES.

Cooling tower. R. A. LEWIS (E.P. 272,325, 29.4.26).—The tower comprises a casing with unobstructed interior and unobstructed large spaces beneath for entry of the air. Vertical sprays of the water to be cooled produce both the upward draught and the necessary surface of water. Horizontal sprays of water, which may be either hot water or water already cooled from the pond below, prevent cross-winds blowing mist out of the air inlets. The tower may be constructed in sections comprising long bays joined at their centres by short bays, so that no part of the interior of the tower is far from an air inlet. B. M. VENABLES.

Absorption refrigerating apparatus. J. O. BOVING (E.P. 271,958, 5.3.26).—An absorption refrigerator of the discontinuous type. B. M. VENABLES.

Mixing liquids for the purpose of carrying out reactions and for producing dispersion systems. J. R. GEIGY A.-G. (E.P. 257,274, 18.8.26. Conv., 22.8.25).—Materials constituting the disperse phase are atomised by a jet of gas under pressure and the spray is passed through a sheet of the dispersion medium.

B. M. VENABLES.

Apparatus for purifying and otherwise treating gases. W. H. TAYLOR and C. SHAW (E.P. 266,097, 2.1.26).—An apparatus for purifying air and other gases consists of a vaned drum rotated by the flow of the gas itself and having its curved surface made of wire mesh or perforated metal. The drum rotates horizontally and contacts with a liquid surface which seals the space below the drum and also maintains the perforations in the drum wet. The gas stream to be cleaned is directed by vanes tangentially against the perforated plate and passes through the moist perforations.

S. PEXTON.

Centrifugal machine. M. L. SANSARICQ (E.P. 272,047, 21.6.26).—The apparatus comprises a number of baskets arranged round a hollow shaft and rotating as a group round the axis of the machine, each one in addition rotating round its own axis, being driven at a variable speed by means of friction and bevel gearing and a second shaft within the hollow shaft.

B. M. VENABLES.

Centrifugal extractors. H. J. PARKER (E.P. 272,368, 2.10.26).—In a centrifugal machine the bowl or basket is directly coupled to a motor beneath it. The weight of the bowl, its contents, and the electrical rotor is taken by a thrust bearing between the bowl and the motor casing. The weight of the whole is taken by a stiff pneumatic diaphragm beneath the motor, so that the bowl can wobble under unbalanced load. An electrical brake is provided which is automatically pulled off just before the motor starts. B. M. VENABLES.

Centrifugal purifying and dehydrating apparatus. EMPSON CENTRIFUGALS, LTD., and S. ALEXANDER (E.P. 272,339, 11.6.26).—In a centrifugal machine as described in E.P. 224,935 (cf. B., 1925, 57) and provided with crater-shaped separating cones, a horizontal annular baffle is inserted in the annular space between the hollow shaft and the crater of the uppermost cone; this baffle is stated to improve the separating action and cause the solid and viscid impurities to pass out continuously with the heavy wash liquid. The baffle may be adjustable.

B. M. VENABLES.

Filters and the like. E. C. HATCHER (E.P. 272,266, 2.3.26).—The filter medium comprises a spiral spring which may be more or less compressed to alter the size of the spaces between the coils. The spring is supported against lateral collapse by ribs on the outer casing and by a ribbed sleeve internally; the latter may be rotated to act as scraper. An alternative construction comprises a filter medium in the form of a clock spring which may be more or less wound up. B. M. VENABLES.

Centrifugal filtering apparatus. A. W. EMPSON (E.P. 271,916, 5.10.26).—In a centrifugal apparatus in which the solids (or heavy liquid) are collected on the outer wall of the rotating bowl and the lighter liquid passes inwards through an edge filter comprising a number of rings of paper compressed together, the filter is constructed of a long ribbon of paper, of small width compared with the radius of the filter and wound edgewise between two perforated cylinders acting as formers. To alter the compactness of the filter while in use, the top is pressed down by an expansible pneumatic tube connected to a pump on the shaft of the machine.

B. M. VENABLES.

Apparatus for spraying or atomising liquids or for disseminating gases. R. A. LEWIS (E.P. 272,003, 20.4.26).—A device in which fluid is admitted at high velocity tangentially to a spiral-shaped chamber and exhausted in the form of spray either axially to the chamber or at another part of the circumference, is constructed with the lining of the spray chamber separable from the main body of the apparatus so that the spiral may be die cast or moulded in such materials as glass, bakelite, or aluminium–silicon alloy (which is electrolytically neutral to cast iron); machining is thus avoided or much simplified. B. M. VENABLES.

Pulveriser. O. A. KREUTZBERG (E.P. 272,111, 21.12.26. Conv., 5.11.26).—A pulveriser with air separation comprises a table which is rotated and on which bear grinding rollers. Air is admitted with a whirling horizontal motion to the grinding zone, which also serves as a first precipitation chamber, and leaves with the ground material upwards through an annular chamber the inner wall of which is composed of adjustable vanes, causing the air to enter horizontally and whirling into another precipitation chamber from which the oversize material drops back to the grinding zone.

B. M. VENABLES.

Tumbling mill. H. W. TITGEN (E.P. 266,664, 8.7.26. Conv., 25.2.26).—The mill comprises a tapered shell within another shell with a reverse taper, both rotating together on a substantially horizontal axis. The goods to be cleaned and the abrasive pass through the inner shell from the small to the large end, at which point the goods pass out of the apparatus, but the abrasive drops through perforations into the outer shell along which it returns to the feed end, where it is picked up by lifting vanes and returned to the inner shell for use again. B. M. VENABLES.

Separating substances such as solids and gases from liquids. GILCHRIST & Co., Assees. of W. C. GRAHAM, H. S. RUMSEY, and A. U. WETHERBEE (E.P. 245,476, 5.1.26. Conv., 5.1.25).—See U.S.P. 1,602,014; B., 1927, 32.

Tubular heat exchanger. H. HILLIER (E.P. 273,450, 26.5.26).

Refrigerating apparatus. SILICA GEL CORP., Assees. of E. B. MILLER and W. L. EDEL (E.P. 260,542 and 264,859, 24.3.26. Conv., 28.10.25).

Separating dust or fume from air and [dust-discharging device in] apparatus of the fabric tube filter type therefor. J. P. VAN GELDER (E.P. 273,563, 22.11.26).

Adsorbent or catalytic masses (E.P. 255,904).—See VII.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Problems encountered in preparing coal for the market. F. S. SINNATT (J.S.C.I., 1927, **46**, 242—250 T).—Various aspects of the relationship existing between the properties of coal seams and the preparation of commercial grades for the market are reviewed. The heterogeneous nature of coal seams is considered, and an attempt made to indicate in general terms the effect this may have on the commercial grades obtained from seams. Sized coals obtained by screening run-of-mine coal exhibit differences not only in caking properties, but also in the amount of ash present and the m.p. of the ash of the coals. The absence of any uniform nomenclature to describe the commercial grades of coal is mentioned, and the necessity for an investigation of the sizes of coal most suited to particular coal seams is suggested. The effect of the size of the coal on the results obtained in the carbonisation of the coal, both in gas retorts and in low-temperature carbonisation, is discussed, and attention is drawn to the associated problem of the influence of the addition of inorganic matter to coal on the mechanics of carbonisation and combustion. In connexion with blending, it is shown that if, for example, equal proportions of two seams are mixed together at a colliery and the blend is then separated into commercial grades by screening, the grades will contain varying proportions of the two seams and will differ in properties, calorific value, etc. according as the seams are distinct in quality and yield varying proportions of the sized coals. The method of testing coals is examined briefly and ash characteristic curves prepared by the Henry tube and the method of specific gravity analysis are compared.

Comparison of processes for cleaning coal. W. R. CHAPMAN and R. V. WHEELER (J.C.S.I., 1927, **46**, 229—238 T).—A number of points are indicated with regard to which it is profitable to compare processes for the cleaning of coal. The theoretical principles upon which eight types of cleaning processes depend are outlined, together with the limitations imposed on them by reason of the principles involved. All of these eight typical processes are compared with respect to seven conditions which, it is suggested, a coal-cleaning plant should be able to fulfil, viz.: (1) there must be differentiation between light and heavy middlings; (2) there should be little mechanical stress on the coal particles; (3) the particles should be in contact with water for a minimum time; (4) fine adjustments should be reduced to a minimum; (5) it should be possible to stop or start the plant rapidly, and efficient washing should obtain immediately on re-starting; (6) there should be efficient operation with an irregular rate of feed and a feed varying in quality or size; (7) there should be a minimum number of accessory appliances such as pumps, motors, elevators, conveyors, and screens. The processes considered are the Draper, Baum, Rheolaveur, and Chance washers, the Deister–Overstrom concentrating table, the Wye and Spiral separators, and froth flotation.

Cleaning of coal, with special reference to pneumatic separation. C. W. H. Holmes (J.S.C.I., 1927, 46, 270—279 t).—The development of pneumatic as opposed to hydraulic classification is discussed, dealing more fully with development from the Sutton, Steele, and Steele pneumatic separator to the "S.J." and the "Wye" type. The laws governing the separation of particles by virtue of the difference in their densities are discussed, and the advisability of applying the mathematics of hydraulic classification to pneumatic separation is questioned. The dry cleaning of coal by pneumatic separation is discussed in relation to the carbonisation industries. Notes are appended describing some of the methods adopted in recent investigations on the process of pneumatic separation which is being carried out by the Birtley Iron Company.

Froth flotation applied to a Baum [coal] washer. W. Guider (J.S.C.I., 1927, 46, 238—242 t).—An investigation of the washing conditions in the Baum apparatus led to the conclusion that fine dirt had no adequate outlet from the washer, and froth flotation was introduced as a means by which the dirt could be discharged from the washer while the fine coal was recovered. The installation of the machine and its effects on the washer are exhaustively described and a detailed table of costs is given. The advantages and disadvantages of froth flotation plant are considered; it is stated that the reduction of ash content in the slack is obtained at the expense of an increase in moisture content.

Float-and-sink testing of small coal. J. L. Thomson and C. N. Kemp (J.S.C.I., 1927, 46, 206—209 t, 312—313 t).—A semi-portable self-contained unit adapted for use in the control laboratory, or on the washing plant itself, is described. The capacity of the float-and-sink vessel may be such as to meet individual requirements, and the separation of the float and sink is facilitated by the special construction of the jig tube within the main container. The removal of each successive float is effected by means of the specially shaped scoop which is introduced after separation, between the float and the sink. The handling of the various solutions is obviated by the employment of compressed air (about 5 lb. per sq. in.) as the means of filling the container, which is emptied by gravitation after the separation of each float. Although the unit is designed to carry out separations through a range of specific gravities, any individual solution is immediately available for a special test for any particular purpose.

Recovery of ammonium thiocyanate in coking. W. Gluud and W. Klempt (Z. angew. Chem., 1927, 40, 659—660).—The results of large-scale experiments on the removal of hydrocyanic acid from coke-oven gases by the thiocyanate process, in which the crude gases are passed through washers in which an aqueous sulphur suspension is circulated, are described. After twelve days, during which 2000—3000 cub. m. of crude gas were treated per hr., the solution attained a strength of 20% by weight of ammonium thiocyanate, the efficiency of extraction of hydrocyanic acid being 98%; only one washer was employed. It is possible to obtain much stronger solutions, but after a strength of 20% has been exceeded the extraction is no longer quantitative. Suggestions are made for possible uses of the thiocyanate on a scale which would absorb the possible production from coke ovens in Germany, estimated at 20,000 tons per annum. S. I. Levy.

Determination of the quality of activated carbons. Z. Vytopil (Z. Zuckerind. Czechoslov., 1927, 51, 511—514).—In examining carbons as to their suitability for use in sugar manufacture, the author determines the water, ash, water-soluble matter, reaction, and the weight of 1 litre of loosely-packed material. The decolorising power is determined by Spörry's equal-effect method (cf. B., 1923, 369 a), the results being compared with Carboraffin or one of the Norit grades as standard. A 4% solution (by vol.) of beet molasses, or better the actual product to be treated in the refinery, is heated with the carbon at 80—90° for 10—15 min. Other determinations which might be made are: adsorption of inorganic substances, alteration of p_H during decolorisation, rate of filtration, and power of regeneration. It is not possible to translate the figures obtained in the laboratory into practice, where a much better decolorising effect may be obtained when the method of filtration through a layer on the filtering surface is applied. J. P. Ogilvie.

Value of low-temperature tar as the carburetting oil for water gas. K. Shimomura (J. Fuel Soc. Japan, 1927, 6, 50—51).—Experiments on an industrial scale have shown that low-temperature tar is as satisfactory for carburetting water-gas as the gas oil usually used for the purpose. Creosote oil, on the contrary, gave poor results. A. B. Manning.

Deposit on an underground gas main. A. Marsden (Gas J., 1927, 179, 219—221).—An 18-in. foul main laid 40 years ago and recently exposed had become covered externally by a crystalline deposit consisting essentially of anhydrous calcium acetate (93%) and containing some butyrate. The calcium had evidently been derived from the marl in which the main was laid, and it is suggested that the acetic and butyric acids have been produced by bacterial action on organic material conveyed by water percolating through the soil. Hot crude gas passed through the main, so that the temperature of the adjacent soil would favour bacterial action. The main was not corroded. A. B. Manning.

Production of liquid fuels from coal. J. G. King (J.S.C.I., 1927, 46, 181—186 t).—The three main lines along which the problem of oil production from coal is being attacked are described and the latest results reviewed. The effect of temperature of carbonisation on the constitution of low-temperature tar is illustrated by experiments carried out at H.M. Fuel Research Station. There are indications that 550—600° is a critical temperature so far as the tar is concerned when external heating is used. At 600° only 8·8% of the tar is saturated. On a large scale the yields of tar are not much greater than 16·5 gals. per ton of coal, and if 50 million tons of coal were carbonised only 150 million gals. of motor spirit and 550 million gals. of tars oils would be available—much less than the country's requirements. The Bergius process offers technically 100—130 gals. of oils per ton of coal or up to

60% by weight of suitable coals. The plant is described and yields are quoted. The catalytic production of alcohols and hydrocarbons from carbon monoxide is showing considerable promise for the future, but so far results are only experimental. The recent work of Fischer and Tropsch in producing petroleum-like hydrocarbons shows particular promise. The catalysts used and the nature of the end products are described.

Products of combustion from coal gas flames. H. Davies and H. Hartley (J.S.C.I., 1927, **46**, 201—206 t).—The iodine pentoxide method was employed to determine the carbon monoxide in diluted products of combustion. Simultaneously the air of the laboratory was also examined. Large volumes were employed and an accuracy of 1—2 parts per million was attained with the apparatus and procedure used. Well-aerated and poorly-aerated Bunsen as well as luminous flames were investigated, and the results of 27 experiments are given. The experimental results indicate that traces of carbon monoxide escape from the combustion zone. On the assumption that only the air theoretically required for complete combustion actually enters into the combustion phenomena, it is calculated that the ratios of carbon monoxide to dioxide in undiluted products of combustion are respectively 0·00024, 0·00034, and 0·0001 for the three types of flames. These ratios correspond to 31, 44, and 13 parts per million of carbon monoxide in undiluted dry products.

Fuel for internal-combustion engines. (Sir) F. L. Nathan (J.S.C.I., 1927, **46**, 211—220 t).—The world's petrol position is discussed and the conclusion is drawn that petrol will continue for a long time to come the principal liquid fuel, but that it will probably be supplemented in due course by a liquid fuel from shale. The possibilities of producing alternative liquid fuels within the Empire are considered, such fuels being benzol and tetralin, both coal distillation by-products, and alcohol from vegetable materials rich in starch, and from the cellulose of plants—a process for this is described. Other sources of alcohol referred to are sulphite waste liquors, ethylene from coal and coke-oven gas, and carbide. The production of liquid fuels by the low-temperature carbonisation and the hydrogenation of coal, as well as their synthetic production from carbon monoxide and hydrogen, is briefly described. Gaseous fuels, mainly coal gas and producer gas, are also referred to.

Separation of the components of petroleum. V. Edge filtration. I. Isolation of waxes from untopped crude oil without pyrolysis. P. F. Gordon and A. C. Marshall (J.S.C.I., 1927, **46**, 304—306 t).—The effect of filtering untopped crude Persian oil through a laboratory model of the Hele-Shaw stream-line filter under varying conditions of temperature and of pack and oil pressure was examined. Black, viscous, asphaltic material can be separated at ordinary temperature; the yield is increased by decrease of temperature. By cooling the crude oil in ice and salt and receiving the filtrate at 11—12° quantities of this residue (d^{22} about 0·899) varying, according to pack and oil pressure, from 1·6 g. to 12·4 g. were removed from 100 c.c. of crude oil (*d* about 0·839). Up to about 60% of white wax could be isolated from these residues. The waxes had different forms, *e.g.*, amorphous brittle masses; amorphous clouds; amorphous flakes; granular clusters; long needles; fibrous flocks, and lustrous fibrous flakes. They had m.p. varying from 67·5° to 76°. One series of filtrates separated into two well-defined layers on cooling—the lower viscous and rich in asphalt, the upper mobile and volatile. Clusters of crystalline wax separated from the upper layer during slow low-temperature evaporation. The physical condition of waxes in crude oil is discussed.

Detection of coal tar and coal tar distillates in petroleum asphalt and natural asphalt. (Miss) J. C. M. Sauerbier (Chem. Weekblad, 1927, **24**, 348—350).—Coal-tar products may be detected by means of Graefe's reaction, which gives better results if diazotised sulphanilic acid is used in place of diazobenzene chloride. The material is boiled with sodium hydroxide solution, the whole filtered when cold, and the test carried out with the filtrate. The proposal of Marcusson to distinguish between coal tar itself and its distillates in admixture with an asphalt by means of the solubility of the latter in light petroleum is found to give no trustworthy results. S. I. Levy.

Volatility and carbonisation of oils for cylinder lubrication. J. W. Donaldson (J.S.C.I., 1927, **46**, 324—327 t).—Examination of three mineral oils—light, medium, and heavy—used for cylinder lubrication and tested under varying conditions of temperature (350—550° F.), varying periods of time (15—60 min.), and over varying surface areas (0·5—2·0 sq. in.), indicated that volatilisation was proportional to the varying times and areas. The character of the residues was determined by the temperature and showed that the light-bodied oil did not carbonise till most of the oil had volatilised and only a small deposit remained, whilst the heavy-bodied oil volatilised slowly, even at high temperatures, and left a large deposit.

Corrosion in petroleum distillation plant. II. M. Freund (Chem.-Ztg., 1927, **51**, 528; cf. B., 1919, 808 a).—The corrosion of the iron valves and condensing columns of the apparatus used in distilling crude petroleum with steam may be due to two causes—the presence of chlorides in the emulsified water in the oil and the production of a large volume of carbon dioxide by partial oxidation of the unsaturated hydrocarbons in the oil. The latter is due to entrapment of air in the steam used or to leakage of air into the apparatus. Thus, in the distillation of crude benzine from Boryslav petroleum at 400°, the uncondensed gases contained as much as 2·5% of carbon dioxide and the issuing water from the apparatus contained 4 kg. of iron per 100 m.^{3} of oil distilled, partly as chloride and partly as ferrous hydrogen carbonate. To avoid these troubles addition of lime or dilute alkali solution is recommended, together with the use of air-free water in the boilers. A. R. Powell.

Burning of mineral oils in wick-fed lamps. J. Kewley and J. S. Jackson (J. Inst. Petrol. Tech., 1927, **13**, 364—397).—The essential features of a good oil include no loss of illuminating power during use and no formation of deposits on the glass. The impurities

likely to occur in kerosenes are sulphur compounds, including sodium sulphonates. The formation of bloom on lamp glasses is caused by sulphur compounds in the kerosene and also by fog or ammonia in the atmosphere. A new chimney accumulates bloom more easily than an old one. Bloom was found to contain organic sulphur acids, ammonium and sodium sulphates, with traces of potassium and calcium. The cooler the chimney the greater is the deposition of bloom, other things being equal. The brighter the flame shortly after lighting the lamp the better is the kerosene for burning purposes. Satisfactory results were obtained with a Weber photometer, whereby a straight-line relationship was observed between height of flame just on the point of smoking, and candle power of that flame. The liquid extracted by liquid sulphur dioxide from Borneo kerosene provided a typical aromatic kerosene; typical paraffinoid and naphthenic kerosenes were also obtained. It was then found that for kerosenes of similar boiling ranges the initial illuminating power is determined by the chemical composition. The higher the boiling range the less is the illuminating power. Viscosities and capillarities of kerosenes were examined in order to investigate the tendency of some kerosenes to give a steadily diminishing flame; in general those with a high viscosity possessed a high capillarity. The tendency of the wick to char appears to be connected with the viscosity and the chemical composition of the oil. It was found that the lighter fractions of the kerosene did not burn first during tests, but that the oil burnt as a whole. The chief cause of diminishing flame size in domestic lamps is the fall in oil level in the reservoir. W. N. HOYTE.

Burning tests of kerosene. W. H. THOMAS (J. Inst. Petrol. Tech., 1927, **13**, 402—409).—In the author's tests 900 c.c. of oil are fed to a special lamp and burnt by means of a flat wick $\frac{3}{4}$ in. wide, the test continuing without attention to the lamp after the first hour, for 24 hours. Flame dimensions are adjusted to 1 in. height, 1 in. wide. The fall in candle power is observed together with oil consumption, amount and type of film on the glass, and condition of the wick. The normal consumption is 27 c.c. per hour. The lamp gives between 5 and 6·5 candle power. W. N. HOYTE.

"Berginisation" and an investigation of hydrocarbon mixtures. I. H. I. WATERMAN and J. N. J. PERQUIN (J. Inst. Petrol. Tech., 1927, **13**, 413—423).—Tests on a large number of hydrocarbons showed that the refractive indices give more accurate information on the purity of the substance than does the dispersivity; the latter constant is, however, of use in identifying the class of hydrocarbon. Bromine values and mol. wt. determinations in many cases gave useful information. In an attempt to crack paraffin wax in the vapour phase the vapours were raised to 450° for 4 seconds. No decomposition was observed. Further experiments in which paraffin wax was heated with aluminium chloride at 200° and then 250° gave yields of 13·9% and 11·2% of gasoline respectively. W. N. HOYTE.

Solubility of oxygen, carbon dioxide, and nitrogen in mineral oil; transfer of carbon dioxide from oil to air. L. S. KUBIE (J. Biol. Chem., 1927, **72**, 545—548).—1 c.c. of a heavy paraffin oil dissolved 0·841 c.c. of carbon dioxide at 24—25°, 0·134 c.c. of oxygen at 28°, and 0·071 c.c. of nitrogen at 22—22·5°, at atmospheric pressure. A saturated solution of carbon dioxide in the oil, when exposed to the air, lost carbon dioxide rapidly from the upper layers, but very slowly from the lower. C. R. HARINGTON.

Development of design of by-product coke oven. R. A. MOTT (Fuel, 1927, **6**, 373—380).

Ammonium chloride from gas liquor. ADOLPHI. —See VII.

Ammonium sulphate plant. ERNST and EDWARDS. —See VII.

Adsorption of activated carbons. SPENGLER and LANDT, also TRAUBE.—See XVII.

PATENTS.

Process and apparatus for testing samples of coal by distillation. R. GEIPERT (E.P. 268,718, 23.9.26. Conv., 31.3.26. Addn. to E.P. 267,082; B., 1927, 516).—To prevent the undue expansion of coal during distillation in the test apparatus, a rod having an enlarged end extends through a gas-tight joint into the distilling chamber and rests on the coal blocks. These blocks are grooved and perforated to facilitate the passage of the gases evolved. A. C. MONKHOUSE.

Coke oven. C. STILL (E.P. 260,974, 20.10.26. Conv., 5.11.25).—The products of distillation from a coke oven are withdrawn through slats, arranged like a Venetian blind, into a hollow space in the door of the oven, and thence to a passage below the sole of the oven. A. C. MONKHOUSE.

Distillation of solid fuel in coke ovens. C. STILL (E.P. 266,584, 15.6.26).—In a coke oven, the distillation products are drawn through a perforated oven sole into a heat-insulated flue. The gas off-take may consist of a channel running longitudinally and centrally beneath the sole of the oven, openings in the channel being arranged to register with corresponding grated openings in the sole, or, alternatively, it may consist of a box-like compartment extending the whole width of the oven and containing two hollow-cast members which support the perforated oven sole. In the latter case, the distillation products are cooled by passing steam through the members in the gas off-take, which steam may then be passed into the crown of the oven. The rich distillation products from the central uncarbonised portions of the charge are drawn off continuously without contacting with the hot crown of the oven. S. PEXTON.

Low-temperature distillation retort. A. V. ABBOTT (E.P. 266,193, 26.6.26).—The retort consists of four co-axial truncated cones revolving in a combustion chamber. In the annular spaces between the two outermost and between the two innermost casings are hermetically-sealed steel flasks containing a fusible metal, *e.g.*, lead. The centre annulus contains the coal undergoing distillation. Coal is fed into the distillation chamber where it is forced by inclined ribs to travel in a circular and longitudinal direction along the outer heating wall until, at a certain position in the rotation, it is caused to fall on to the inner heating wall, which is also provided with guiding ribs so staggered in relation to the

others that the progress of the coal is retarded. The carbonised fuel falls over an inclined apron which revolves with the retort and projects into the gas-collecting chamber. A condensing coil is built into this chamber and tar condensed on its surface is drained away.

S. Pexton.

Distillation of solid carbonaceous substances. T. M. Davidson (E.P. 273,389, 30.3.26).—Carbonaceous substances, such as coal, are carbonised in a vertical retort with a taper of about 1 in 12, and such that on periodically discharging part of the coke at the base, the remainder of the charge is lowered as a coherent mass, leaving an interstitial space between the retort wall and the charge, into which a fresh charge of the substance is introduced. The fresh charge is thus heated in a thin layer from both sides, heat transmission and carbonisation being thereby rapidly effected.

A. B. Manning.

Distillation of coal [of high sulphur content]. R. G. Griswold, Assr. to Doherty Research Co. (U.S.P. 1,635,667, 12.7.27. Appl., 29.4.22).—The coal is heated by direct heat transfer from a hot gas of low calorific value in such a way as to produce coke and a rich gas of high sulphur content. Part of the coke is burnt and the hot gaseous products are used to heat the remainder to incandescence, steam being then passed through the latter to generate the gas of low calorific value used for heating a fresh charge. The rich gas is purified and passed through the remaining incandescent coke, the sulphur content of which is thereby further reduced. A. B. Manning.

Destructive hydrogenation of coal, tar, mineral oils, and the like. I. G. Farbenind. A.-G. (E.P. 254,713, 28.6.26. Conv., 2.7.25).—Gases containing hydrocarbons and their derivatives prepared by the hydrogenation of carbonaceous substances are further treated with oxygen or gases richer in oxygen than air in the presence of catalysts, *e.g.*, nickel precipitated on magnesia, iron alloys, molten iron. The hydrocarbons are converted into carbon monoxide and hydrogen, water vapour and/or carbon dioxide being added, if required, for the conversion. Hydrocarbons such as ethane etc. may be separated from the initial gases by cooling or by adsorbents. A. C. Monkhouse.

Vertical retort for the extraction of oil from bituminous solid fuels such as shale, coal, peat, and the like. G. Menell (E.P. 272,766, 20.11.26).—The retort consists of a vertical, flattened, narrow shaft with sloping plates arranged alternately on opposite sides of the retort wall to give a zig-zag course to the fuel. The retort is divided into two chambers by a rotary valve, the quantity of material discharged being controlled by similar valves on the charging and discharging ends. The products of distillation are withdrawn from each chamber through outlets which slope upwards from beneath the plates. The retort is heated externally, or internally by superheated steam or gas admitted through pipes beneath the sloping plates.

A. C. Monkhouse.

Apparatus for the distillation of fuel, roasting ores, and like heating purposes. J. Plassmann (E.P. 254,697, 17.6.26. Conv., 30.6.25; cf. E.P. 240,800; B., 1926, 228).—The annular heating chambers or cells are built up from segments of refractory material. The adjacent superposed segments are offset in relation to each other and are so shaped that the gases follow a zig-zag path round the apparatus. There is a central heating-gas and mixing shaft in the upper part of which is a heat-equalising accumulator. The apparatus may be worked at a temperature high enough for the production of metallurgical coke. R. A. A. Taylor.

Low-temperature carbonisation of coals etc. K. M. Simpson (E.P. 273,935, 22.9.26).—A series of receptacles is affixed to an endless carrier. While they are passed in the direction in which they are inverted, they traverse a zone in which they are heated sufficiently to carbonise a charge of finely divided carbonaceous material, which is fed into them when they pass round into the upright position and on through a distilling zone. They are re-inverted at the discharge end for the carbonised material, and necessary steps are taken to recover the distilled products. R. A. A. Taylor.

Manufacture of illuminating and industrial gases. H. Nielsen and B. Laing (E.P. 272,822, 1.11.26).—A rotating inclined producer is used for the manufacture of water-gas. Gas and air for combustion are admitted at one end and, after combustion, pass through chequer brickwork in channels surrounding the producer. When the requisite temperature is attained, steam, preheated at 1000° or above, is admitted in reverse direction through the chequer brickwork and passes through the semi-coke in the producer. The resulting gases are withdrawn from the fuel inlet end of the producer, their sensible heat being further utilised in retorts. The heating value of the gases may be increased by passage through heated chequer brickwork for the purpose of cracking the tarry vapours.

A. C. Monkhouse.

Manufacture of gas. Humphreys & Glasgow, Ltd., and J. C. Stelfox (E.P. 266,108, 20.1.26).—In the manufacture of gas by the complete gasification of coal in a single generator, the mixed coal gas and water-gas produced is of low calorific value. It is proposed to limit the quantity of water-gas by withdrawing part of the coke from the carbonising zone and thus make a high-grade gas together with some coke. In a recess, built round the generator at a level above the blast offtake, is mounted a rotary ring carrying doors hinged on a vertical axis. As the ring rotates in one direction the doors engage in and extract part of the well-carbonised periphery of the charge, which is diverted into suitable chambers ready for discharge. When the direction of rotation is reversed the hinged doors close and disengage from the coke, extraction thereby ceasing. The quality of gas made can be varied by adjusting the proportion of coke discharged without resorting to carburetting. S. Pexton.

Treatment of [fuel] gases with liquids. E. L. Pease (E.P. 273,103, 7.7.26).—A suspension of calcium sulphate is used to wash the gas, when, with ammonia and carbon dioxide present, ammonium sulphate and calcium carbonate are formed. The spent liquor is then heated at a higher temperature (80—90°) and preferably at a reduced pressure. The reaction is reversed and the

ammonia and calcium sulphate are regenerated, the former being absorbed by a suitable acid agent.

R. A. A. Taylor.

Purification of gas. Humphreys & Glasgow, Ltd. From E. J. Brady (E.P. 266,586, 18.6.26).—Gas to be purified is passed upwards through a vessel of gradually increasing cross-section against a cloud of finely-divided purifying agent (*e.g.*, hydrated iron oxide) which gravitates down the vessel. The velocities of the gas and of the purifying agent progressively decrease, but their relative velocities at various stages are fairly constant. S. Pexton.

Reducing to carbon monoxide the carbon dioxide content of gases. F. M. Wiberg (E.P. 266,729, 23.2.27. Conv., 27.2.26).—A charge of carbonaceous material is partially burnt by means of air and the gas containing carbon dioxide, admitted simultaneously or alternately; further quantities of carbon dioxide are reduced by contact with a fresh portion of the charge heated electrically. The method may be utilised by alternating the processes of passing air through one and generating water-gas in the other of a pair of water-gas producers, and then passing the mixed gases through a chamber containing electrically-heated carbonaceous material.

R. A. A. Taylor.

Manufacture of motor fuel. I. G. Farbenind. A.-G. (E.P. 251,969, 27.4.26. Conv., 8.5.25).—To minimise "knocking" without substantially reducing the calorific power of the fuel, anhydrous methyl alcohol, either alone or mixed with higher alcohols or ketones, is added to gasoline. S. Pexton.

Production of liquid hydrocarbons and other organic substances from heavy organic materials. G. Patart (E.P. 249,519, 15.3.26. Conv., 21.3.25).—The liquids or gases resulting from high-pressure syntheses, *e.g.*, the manufacture of methyl alcohol, are utilised at their respective pressures and temperatures for the treatment of carbonaceous solids or liquids, *e.g.*, coal, peat, heavy petroleum oil, in suitably designed digesters. Distillation or hydrogenation takes place and the products are recovered. Catalysts may be used in the digesters and the gas mixtures required in the initial operation prepared from the residue of the products by incomplete carbonisation. A. C. Monkhouse.

Purification of hydrocarbons. I. G. Farbenind. A.-G. (E.P. 255,905, 26.7.26. Conv., 27.7.25).—High-boiling hydrocarbon oils are treated with mixtures of methyl alcohol and low-boiling aromatic or naphthenic hydrocarbons. The impurities in the raw material are dissolved and the purified oil, not being miscible with the washing material, can be separated.

R. A. A. Taylor.

Converting high-boiling hydrocarbons, which have been freed from the substances soluble in liquid sulphurous acid, into low-boiling hydrocarbons by means of aluminium chloride. Allgem. Ges. für Chem. Ind. (E.P. 273,999, 11.3.27. Conv., 17.9.26. Addn. to E.P. 272,433, B., 1927, 595).—The residual oils, before the renewed treatment with aluminium chloride, are treated with concentrated sulphuric acid instead of with sulphur dioxide.

R. A. A. Taylor.

Refining mineral oils and/or their distillates. Burmah Oil Co., Ltd. From J. P. Fraser (E.P. 273,351, 25.1. and 21.9.26).—Mineral oils, or their distillates, are refined by extraction with phenol, or a mixture of phenol and cresylic acid, containing 5—15% of water. The oil is separated and washed with a solution of caustic alkali, from which any phenol is subsequently recovered by acidification. A part of the mineral oil dissolved in the phenol is recovered by partial dilution of the latter with water and separation of the oily layer so produced. The phenol and dissolved substances are then separated by agitating the mixture with a large volume of water at a temperature between 90° and 100°, or, in refining low-boiling fractions, by distilling to 120°. A. B. Manning.

Cracking of petroleum oils and carburetting of water-gas. Petroleum Chemical Corp., and E. P. Stevenson (E.P. 273,781, 24.3.26).—A vaporous distillate from petroleum is cracked in a tubular retort; the gaseous product is stripped by condensation of heavier residues and motor spirit and by removal of hydrocarbons of three or more carbon atoms, especially the unsaturated hydrocarbons liable to form gummy deposits. The remaining rich gas is mixed with a lean gas, *e.g.*, water-gas. The production of olefines is promoted by inhibiting any exothermicity in the cracking process. The heating may be arranged in zones according to the temperature in the preceding zone. The olefine fractions and spirits in an oil are developed by cracking the vapours which distil off only up to 400°, the non-volatile residue being removed. Before the residual gas is used for carburetting it may be further stripped by refrigeration or oil-scrubbing. Before or after mixing with the lean gas, any remaining condensible constituents may be fixed by heating the gas to a temperature higher than the cracking temperature.

R. A. A. Taylor.

Gas-testing apparatus with pressure gauges, more particularly flue-gas testers. Siemens & Halske A.-G. (E.P. 262,092, 16.11.26. Conv., 27.11.25).

Ferric sulphate (E.P. 273,883).—See VII.

Electrical purification of gases (G.P. 439,693).—See XI.

III.—TAR AND TAR PRODUCTS.

Variation of Hutchinson consistency of tars with temperature. H. M. Spiers (J.S.C.I., 1927, **46**, 329—330 T; cf. B., 1927, 38.)—The criticisms by Mallison and Soltau (B., 1927, 517) are met by calculations made from the results quoted by these authors. The mathematical relationship previously proposed is shown to hold good over intervals of temperature as large as 10° C. It is pointed out, however, that one or more transition points may occur where sudden changes in the value of the constant are encountered due to alterations in the physical nature of the tar possibly caused by crystallisation of naphthalene, deposition of waxy matter, or change in the state of aggregation of the dispersed colloids.

Asphalt-like substances in coal tar. G. Šebor (Petroleum, 1927, **23**, 890—897).—The methods used for determining "asphalt" in mineral oils give widely

different results when applied to a coke-oven tar. From a consideration of the behaviour of the tar on admixture with different solvents the following procedure is recommended for separating the "asphalt" from the residual oil and determining the content of each: 1 g. of tar is dissolved in 10 c.c. of carbon disulphide at the ordinary temperature and 100 c.c. of methyl alcohol are added. After 2 hrs. the clear liquid is decanted through a filter and the residue washed with methyl alcohol until the filtrate is colourless. The residue in the flask and on the filter is dissolved in benzene, transferred to a basin, and after evaporation of the benzene weighed. The properties of both the asphalt and the residual oil have been fully investigated. The asphalt content of a number of coke-oven tars varied from 16·5 to 24·5%. A marked increase in the value was observed on heating the tar to 200°. The method can be applied to other types of tar, pitches, etc. A. B. MANNING.

Low-temperature tar for carburetting water-gas. SHIMOMURA.—See II.

Coal tar etc. in asphalt. SAUERBIER.—See II.

PATENTS.

Distillation of coal tar, tar oils, and similar liquids. A. MEIRO (E.P. 267,074, 15.6.26. Conv., 2.3.26).—After a preliminary dehydration, tar is fed into a still where it is kept in constant agitation by stirrers. The distillation is carried out under pressure and the products pass into a series of dephlegmators, the final gas after purification being returned to the still. The flue gases from the still pass through flues to the dephlegmators, each of which is independently controlled. The liquid fractions condensed pass to coolers and receivers. A. C. MONKHOUSE.

Separation of water from mixtures of steam and vapours of benzene, toluene, and like hydrocarbons. C. COOPER, D. M. HENSHAW, and W. C. HOLMES & Co., LTD. (E.P. 273,224, 16.12.25).—Mixtures of the vapours of water, benzene, toluene, etc., and in particular the mixed vapours arising from the steam-distillation of the wash oil used in the recovery of benzene from coal gas, are freed from water by treatment with a suitable absorbing liquid, *e.g.*, a concentrated solution of calcium chloride. The vapours are brought into contact with the absorbing solution in a column in which the solution runs counter-current to the vapours; the latent heat of condensation developed in the column is utilised for the re-concentration of the absorbing solution. A. B. MANNING.

Extraction of crystals from anthracene, naphthalene, and the like distillates of coal tar. A. MEIRO (E.P. 255,429, 15.6.26. Conv., 15.7.25).—Naphthalene, anthracene, etc. are separated from the distillate by mixing with the fraction a solvent of b.p. below 100°, *e.g.*, petrol, benzol, light petroleum, carbon disulphide, etc. The mixture is cooled in heat-insulated vessels and, after crystallisation, is passed through a filter at the bottom of the vessel, compressed air being used to separate further oil and solvent from the crystals. After washing with water they are removed and dried. The solvent is recovered from the filtrate by distillation. A. C. MONKHOUSE.

Furnace for heating tar and the like. R. and A. B MACINTOSH (E.P. 273,890, 24.6.26).

Hydrogenation of tar (E.P. 254,713).—See II.

Hydrocarbons (E.P. 255,905).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Synthesis of *iso*violanthrone (*iso*dibenzanthrone). C. MARSCHALK (Bull. Soc. chim., 1927, [iv], **41**, 706—709).—Oxidation of dibenzoylperylene with manganic sulphate or manganese dioxide in sulphuric acid solution at — 10° to — 15° in presence of boric acid affords *iso*violanthrone, which, by treatment with chlorine in nitrobenzene solution at 60°, yields a chlorinated *iso*violanthrone affording dyeings which are brighter and 50% stronger than those of Brilliant Indanthrene Violet RR. Oxidation at room temperature affords a hydroxylated vat dye forming a bluish-violet paste which dyes cotton in olive shades, turning blue with acids. Methylation of this product with methyl sulphate in nitrobenzene in presence of sodium carbonate gives a bluish-green vat dye dyeing cotton in blue shades fast to chlorine and washing. Substituted *iso*violanthrones can be similarly prepared from the corresponding diketoperylenes. R. BRIGHTMAN.

Azoic colours. HIGGINS.—See VI.

PATENTS.

Preparation of azo dyes. I. G. FARBENIND. A.-G. (F.P. 609,136, 12.1.26. Conv., 13.1.25).—Diaryl sulphones which contain in separate nuclei a primary amino-group and an *o*-hydroxy-carboxylic acid grouping are diazotised and coupled with coupling components to give after-chrome dyes for wool, which also yield chrome printings on cotton fast to soap and chlorine. After-chroming does not appreciably change the direct shade on wool. 4-Amino-4′-hydroxy-2 : 3′-dicarboxydiphenyl sulphone, prepared by reducing the condensation product from 5-sulphinosalicylic acid and 2-chloro-5-nitrobenzoic acid, is diazotised and coupled with β-naphthol-8-sulphonic acid for an orange-yellow. 2-Amino-4′-hydroxy-3′-carboxy-4-sulphodiphenyl sulphone, obtained by reduction of the condensation product of 5-sulphinosalicylic acid with 4-chloro-3-nitrobenzenesulphonic acid, is diazotised and coupled with 2-methylindole (yellow), 4-nitro-*m*-phenylenediamine (reddish-yellow), β-naphthol (orange), 4-methoxy-*m*-toluidine, further diazotised and coupled with salicylic acid (reddish-brown), H-acid and further coupled with 2 : 4′-diazodiphenylazosalicylic acid (green), γ-acid (bluish-red). C. HOLLINS.

Manufacture of preparations of diazo salts for dyeing and printing. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 246,181, 19.1.26. Conv., 19.1.25).—Aryldiazonium neutral 1 : 5-naphthalenedisulphonates made from non-sulphonated arylamines are relatively stable, crystallise well from their aqueous solutions, are sufficiently soluble in water (often more soluble in presence of disodium 2 : 7-naphthalenedisulphonate or benzenesulphonate), and give clear shades when coupled on the fibre with, *e.g.*, 2 : 3-hydroxynaphthoic arylamides. The presence of negative groups in the arylamine is advantageous,

and nitrated aminophenol ethers are particularly suitable. C. HOLLINS.

Manufacture of diazo salts and of preparations therefrom. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 269,212, 19.1.26. Conv., 19.1.25).—In place of the neutral 1 : 5-naphthalenedisulphonates of unsulphonated diazonium compounds (cf. preceding abstract) the acid salts are used. These are obtained by adding to a solution of the diazo compound in strong mineral acid a solution of 1 : 5-naphthalenedisulphonic acid or its soluble salts. The products crystallise well, are soluble in water, and are usually more stable than the neutral disulphonates. They may be used alone or mixed with dry sodium carbonate etc. The process is especially suitable for diazotised nitrated aminophenol ethers (*e.g.*, 5-nitro-*o*-anisidine). C. HOLLINS.

Manufacture of dry diazo preparations. I. G. FARBENIND. A.-G., Assses. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 246,870, 30.1.26. Conv., 30.1.25).—Moist or dry diazo salts (especially the zinc chloride double salts or the 1 : 5-naphthalenedisulphonates) are mixed with partly dehydrated alum (containing, *e.g.*, $8H_2O$) or, better, with partly dehydrated aluminium sulphate (containing, *e.g.*, $9H_2O$) to give completely dry, stable preparations, to which may be added diluents or other auxiliary substances ordinarily used. The process is particularly suitable for nitrated diazo compounds. C. HOLLINS.

Manufacture of diazo preparations. A. G. BLOXAM. From I. G. FARBENIND. A.-G. (E.P. 273,352, 25.1.26).—The solubility of dry, stabilised diazo preparations is increased by addition of appropriate metal (sodium) salts of benzene-sulphonic and -disulphonic acids, phenylmethanesulphonic acid, tetralenesulphonic acids, naphthalene-1 : 6-, -1 : 7-, and -2 : 7-disulphonic, -trisulphonic, and -tetrasulphonic acids. Neutral diazonium 1 : 5-naphthalenedisulphonates and diazotised aryl and aralkyl ethers of *o*-aminophenols are excluded. Sodium 1 : 6-naphthalenedisulphonate is added to the zinc chloride double salt of α-diazonaphthalene, or to *p*-nitrodiazobenzene *p*-chlorobenzenesulphonate, or to 4-chloro-2-diazotoluene hydrogen 1 : 5-naphthalenedisulphonate ; sodium naphthalene-tri- and -tetrasulphonate mixture is added to 4-chloro-2-nitrodiazobenzene zinc chloride double salt ; sodium 2 : 7-naphthalenedisulphonate to the zinc chloride double salt of tetrazotised dianisidine. A mixture of moist diazobenzene-*p*-sulphonic acid with sodium 1 : 5-napthalenedisulphonate may be dried at 40—50° and ground without danger. C. HOLLINS.

Stable diazo preparations. I. G. FARBENIND. A.-G., Assees. of K. SCHNITZSPAHN (G.P. 438,743, 13.2.25).—A mineral salt of a primary arylamine, with or without excess of the mineral acid, is mixed with a metal arylsulphonate and any desired dye-bath assistants, and finally with dry nitrite. By dissolving in water or dilute acid a diazo compound is produced. C. HOLLINS.

Vat dyes of the dibenzanthrone series. BADISCHE ANILIN U. SODA-FABR. (F.P. 607,135, 28.11.25. Conv., 13.12.24).—Alkoxy-derivatives of 3-chlorobenzanthrone are treated with alkaline condensing agents. 3-Chloro-2-methoxybenzanthrone, prepared by chlorination of 2-methoxybenzanthrone or by methylation of the chlorohydroxy-compound, when heated with alcoholic potassium hydroxide yields a greenish-blue vat dye. C. HOLLINS.

Manufacture of halogenated benzanthrone derivatives containing sulphur. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 249,890, 29.3.26. Conv., 27.3.25).—Benzanthronyl mercaptan, sulphide, or disulphide (cf. E.P. 238,574 and 256,059 ; B., 1925, 876 ; 1926, 867) is treated with a halogenating agent, *e.g.*, the mercaptan is brominated in chlorosulphonic acid at — 5°, the sulphide is chlorinated with sulphuryl chloride in nitrobenzene at 50° to a dichloro-compound, or brominated in chlorosulphonic acid at —5° to mono- and di-bromo-compounds. The products are intermediates for vat dyes (cf. following abstract). C. HOLLINS.

Manufacture of condensation products of the benzanthrone series and of vat dyes containing nitrogen. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 249,891, 29.3.26. Conv., 27.3.25).—Halogenated benzanthronyl mercaptans, sulphides, or disulphides (cf. preceding abstract) are condensed with amines or amides in presence of an acid-binding agent, and the products are fused with alkali (preferably alcoholic) to give vat dyes. From bromobenzanthronyl sulphide condensed with α-aminoanthraquinone and fused with alcoholic alkali at 160—180° a navy-blue vat dye is obtained. Dibromobenzanthronyl sulphide and α-aminoanthraquinone give a bluish-grey, dibromobenzanthronyl sulphide and benzamide a blue, bromobenzanthronyl mercaptan and α-aminoanthraquinone a blue-black. C. HOLLINS.

Vat dyes of the anthraquione series. FARBENFABR. VORM. F. BAYER & Co. (Swiss P. 115,114, 17.2.25).—5 : 5′-Dibenzoyldiamino-1 : 1′-dianthraquinonylamine is treated with concentrated sulphuric acid and then oxidised (*e.g.*, with oleum containing 40% of free sulphur trioxide) to give a yellow-orange vat dye. C. HOLLINS.

Electrolytic desulphonation of anthraquinonesulphonic acids. BRITISH DYESTUFFS CORP., and A. J. HAILWOOD (E.P. 273,043, 15.4.26).—Electrolytic reduction of salts of sulphonated anthraquinones in alkaline solution in a divided cell (a porous pot, or an impermeable division dipping into a mercury cathode) leads to oxanthrols or oxanthrolsulphonic acids, one or more α-sulphonic groups being removed. A current density of 2—4 amp./dm.² is used. Air should be excluded to prevent re-oxidation. After the reduction and desulphonation, the product may be oxidised by a current of air to anthraquinone or anthraquinonesulphonic acids. 1 : 5- and 1 : 8-anthraquinonedisulphonic acids give oxanthrol ; the 1 : 6- and 1 : 7-acids give oxanthrol-2-sulphonic acid and, by subsequent oxidation, anthraquinone-2-sulphonic acid, isolated as its α-naphthylamine salt. C. HOLLINS.

Manufacture of halogenated dibenzpyrenequinones. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 249,147, 12.3.26. Conv., 14.3.25).—2 : 6-Dihydroxy-1 : 5-diaroylnaphthalenes are treated with a phosphorus halide,

preferably in a solvent or diluent (*e.g.*, chlorobenzene), and then cyclised with concentrated sulphuric acid at 80—90°. From 2 : 6-dihydroxy-1 : 5-dibenzoylnaphthalene and phosphorus pentachloride an intermediate is obtained which is converted by sulphuric acid into 1 : 6-*dichloro*-3 : 4 : 8 : 9-*dibenzpyrene*-5 : 10-*quinone*, a yellow vat dye. C. Hollins.

Manufacture of [quinone vat] dyes. Soc. Chem. Ind. in Basle (Ges. für Chem. Ind. in Basel) (E.P. 258,563, 7.8.26. Conv., 16.9.25. Addn. to E.P. 237,375; B., 1925, 876).—In place of the sulphur dihalide of the prior patent, a solution of halogen in sulphur dihalide, approximating to SX_3 or SX_4, is made to react with a 2 : 5-diaryldiamino-*p*-benzoquinone. 2 : 5-Dianilino-*p*-benzoquinone gives, with sulphur "trichloride" (1 mol.) in nitrobenzene at 5°, a product which dyes wool yellowish-olive from a hyposulphite vat. At 100° a dark olive, and at 200° a black-brown dye results. With 2 mols. of "trichloride," brown (15°), olive (100°), and blue-grey (200°) dyes are obtained. With 3 mols. of "trichloride," brown, red-brown, and blue-grey dyes are formed; with 4 mols., brownish-yellow and dark violet-brown; with 2 mols. of "tetrachloride" at 100°, a red-brown dye is obtained. 6-Chloro-2 : 5-dianilino-*p*-benzoquinone with 1 mol. of "trichloride" gives, at 5° a bright brown dye; at 100° an orange dye; and at 200° a pure grey. Redder products result when 3 mols. of "trichloride" or 1—2 mols. of "tetrachloride" are used at 100°. A blue is obtained by the action of 2 mols. of "trichloride" on 2 : 5-di-α-naphthyldiaminobenzoquinone. C. Hollins.

Preparation of compounds of the indigo group. I. G. Farbenind. A.-G., Assees. of K. Schirmacher and A. Wolfram (G.P. 438,841, 8.8.24).—Indigo etc. is condensed with ketones (acetone, ethyl acetoacetate) in the presence of alkali and the product precipitated, if desired, with acid. The preparation and vatting of the product may be performed in one operation.
C. Hollins.

Preparation of aromatic amines and derivatives. [Separation of mono- and di-alkylarylamines.] British Dyestuffs Corp., R. W. Everatt, and E. H. Rodd (E.P. 273,923, 25.8.26).—The mixture of mono- and di-alkylated arylamines (which may be present in any proportions) is treated with carbonyl chloride in presence of water below 15°. If the proportion of tertiary base is insufficient to combine with all the hydrogen chloride liberated, alkali is added gradually during the phosgenation. The tertiary base is dissolved in dilute acid and the carbamyl chloride of the secondary base is filtered off, and either hydrolysed to give pure secondary base, or condensed with a further quantity of secondary base to give the carbamide. C. Hollins.

[Manufacture of] dye intermediates. J. Thomas, H. A. E. Drescher, and Scottish Dyes, Ltd. (E.P. 273,347, 24.12.25).—Halogenated *o*-benzoylbenzoic acids, excluding *o*-(4-halogen-3-nitrobenzoyl)benzoic acids, react at about 180° with alkali sulphites to give sulphobenzoylbenzoic acids, which are then cyclised with 5% oleum at 90° to anthraquinonesulphonic acids. Thus, *o*-(4-chlorobenzoyl)benzoic acid is converted into anthraquinone-2-sulphonic acid; chlorotoluylbenzoic acid (from *p*-chlorotoluene and phthalic anhydride) into 1-methylanthraquinone-4-sulphonic acid; 3 : 4 : 5 : 6-tetrachloro-2-benzoylbenzoic acid into a dichloroanthraquinonedisulphonic acid; chlorotoluylbenzoic acid (from *o*-chlorotoluene) into a methylanthraquinonesulphonic acid; 3 : 6-dichloro-2-benzoylbenzoic acid into 1-chloroanthraquinone-4-sulphonic acid. C. Hollins.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Polysaccharides. XXXVI. Enzymic degradation of viscose silks. P. Karrer and P. Schubert (Helv. Chim. Acta, 1927, **10**, 430—440; cf. B., 1926, 945).—In 30 samples of viscose silk, precipitated under different conditions, parallelism is observed between (*a*) regularity of cross-section, (*b*) affinity for a direct dye (Chloramine Pure Blue), (*c*) lustre, and (*d*) degree of hydrolysis by cellulose. Silks from a sulphuric acid bath containing no salts are dull and woolly, round and regular in cross-section, readily dyed, and easily fermented. The substitution of part of the acid in the spinning bath by salts (magnesium, sodium, and ammonium sulphates) leads to increased lustre, ellipsoidal cross-section with numerous indentations, loss of affinity for the dye, and much decreased attack by the enzyme; there is no measurable increase in ash content due to adsorbed salts. Viscose, coagulated in an ammonium sulphate bath and fixed either with sulphuric acid, or with sodium sulphate and sulphuric acid, gave round, regular threads equally attacked by cellulase.
C. Hollins.

Recent developments in the cellulose industry. C. J. J. Fox and L. Hall (J.S.C.I., 1927, **46**, 281—284 T).—A survey of recent developments of the industrial application of cellulose and ligno-cellulose in the textile, paper, and timber trades, the relative merits of esters and ethers, the finishing and dyeing qualities of artificial silks, the uses of cellulose films, plastic masses, and lacquers, and the products of cellulose by decomposition, including bacterial decomposition.

Wood and cellulose mucilage. C. G. Schwalbe (Papierfabr., 1927, **25**, 481—485).—For the production of mucilage by mechanical means, wet (especially green) wood is superior to dry, and material rich in hemicellulose and pectin to that containing much lignin. The purer the cellulose, the more difficult is its conversion into mucilage, and such conversion is favoured by the loosening of the structure by swelling. In pulp manufacture, its formation is promoted by excessive cooking or bleaching, by bacterial action, or by the use of certain salts (*e.g.*, lithium chloride and calcium thiocyanate). The mucilage mechanically produced in the hollander is chemically modified, compared with the original material. It has an enhanced copper number (Schwalbe), and a characteristically high hygroscopicity, which is dependent on the degree and duration of drying, whilst its dissolution in 16% sodium hydroxide, with reprecipitation and measurement of the amount of deposit, affords a means of controlling the usual test for the degree of grinding. The most important property of the mucilage for paper making is its capacity for welding in the moist condition, which is favoured by severe pressing of the material and by drying it under conditions which avoid

loosening of the fibre felt (best by heating both sides of the sheet equally and simultaneously by means of radiant heat), increased strength with low shrinkage being thus obtained. Next to the cementing of the fibres by the mucilage, the felting of the fibres is of importance, those which show the most pronounced crimping yielding the strongest paper. B. P. RIDGE.

Determination of mechanical wood in paper by the phloroglucinol method. KORN (Zellstoff u. Papier, 1927, 7, 315—319; cf. B., 1907, 942; 1922, 806 A).—An examination of this method confirmed in the main the accuracy of manipulative details. The absorption of phloroglucinol is not, however, a constant, but increases with the time, but if a treatment of 24 hrs. is allowed, a little more or less introduces only an insignificant error. The question of utility depends essentially on the validity of the factors employed for calculating the results. These would require adjustment according to the purity of the phloroglucinol reagent employed. The author gives mean factors of 6·48 for mechanical pulps and 1·21 for sulphite pulps, as compared with 7·84 and 1·34 found by Krull and Mandelkow. The individual values for mechanical pulp diverge far less from the mean than those for the chemical pulps, which vary with the degree of digestion. When the phloroglucinol values of the ingredients of the paper are ascertainable, the results of the determination are remarkably accurate and the method would be valuable in paper-mill control. When, however, the original ingredients are unobtainable and mean values have to be employed, the error due to divergences from the mean values may amount to 8%. This error is larger than that attendant on microscopical evaluation when relatively small quantities of either ingredient are present, but between the limits of 40 and 70% of mechanical wood the errors in microscopical estimation are very much greater and in such cases the chemical method may be recommended. J. F. BRIGGS.

Manufacture of fibrous cellulose. J. L. A. MACDONALD (J.S.C.I., 1927, **46**, 251—261 T).

Bronzes for cellulose works. RAUCHBERG. **Behaviour of bronze in cellulose bleach liquors.** HEIKE and WESTERHOLT.—See X.

Production of fat by penicillium. BARBER.—See XII.

PATENTS.

Manufacture of filaments of artificial silk or the like from viscose. COURTAULDS, LTD., W. H. GLOVER, and G. S. HEAVEN (E.P. 273,386, 29.3.26).—The lustre of viscose artificial silk filaments is reduced by incorporating with the viscose before spinning a small quantity of a high-boiling petroleum, of petroleum jelly, or a mixture of both (*e.g.*, 0·1—0·5% of jelly). Part of the petroleum is persistently retained by the finished fibres. B. P. RIDGE.

Production of benzylcellulose. PATHÉ CINÉMA (ANC. ETAB. PATHÉ FRÈRES) (F.P. 615,349, 24.9.25).—The mixture of benzyl alcohol, benzylcellulose, unaltered benzyl chloride, sodium chloride, and water obtained by the action of benzyl chloride on alkali-cellulose is thoroughly mixed with castor oil (approx. 2·5 litres to every 500 g. of cotton), and sufficient methyl alcohol is added to dissolve the castor oil and benzyl alcohol. Benzylcellulose separates as an impalpable powder readily washed free from sodium chloride with water. Castor oil may be replaced by other oils that do not dissolve benzylcellulose and are miscible with benzyl alcohol and the wash alcohol. S. S. WOOLF.

Rotary pump suitable for feeding viscose. A. L. and G. E. LUDWIG (GEBR. LUDWIG) (E.P. 273,536, 1.10.26. Conv., 28.7.26).

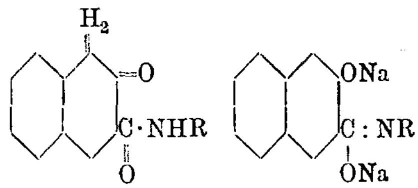
Drying machines for loose textile yarns and fabrics and other materials. T. E. WOOD and H. HEYMANN (E.P. 273,785, 31.1.27).

[Machine for] the manufacture of yarn or thread. A. N. SHELDON (E.P. 273,808, 7.4.26).

[Mechanism for controlling] the washing, scouring, and the like treatment of woven and other fabrics. P. K. WHITEHEAD and A. B. HENSHILWOOD (E.P. 273,854, 29.4.26).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Azoic colours. E. B. HIGGINS (J. Soc. Dyers and Col., 1927, **43**, 213—219).—The "fading" or loss of coupling power on exposure to the air of hanks dipped in a solution of a 2 : 3-hydroxynaphthoic arylide in sodium hydroxide and Turkey-red oil is due to atmospheric carbon dioxide, which promotes the formation of the non-coupling free arylide, (I), the coupling sodium salt possessing the enolic structure (II).



(I) (II)

Oxidation plays no part, a thoroughly good shade being obtained from a Brenthol A.S. bath (2 : 3-hydroxynaphthoic anilide) made up with 5-vol. hydrogen peroxide in place of water. The protective action of formaldehyde is not due to the aldehyde group as such. Sodium sulphite, formate, sulphide, phosphite, hydroxylamine, acetaldehyde, dextrose, and a mixture of sodium formate and sodium hydrogen sulphite are ineffective in preventing "fading," but sodium hydroxide and sodium carbonate are effective, the time of resistance to fading being proportional to the alkali used. Even in the presence of formaldehyde "fading" may occur on prolonged exposure (2 hrs.) or in air of increased acid content. Further evidence in favour of the above view is afforded by the observation that by impregnating hanks in solutions of the anilide in Westron, pyridine, ammonia, and caustic soda, respectively, the depth of shade obtained on development is proportional to the hydroxyl concentration of the solvent. The action of excess of acetyl chloride on 2 : 3-hydroxynaphthoic anilide at 100° affords the brick-red O-*acetyl* derivative, m.p. 130° (decomp.), from which the acetyl group is eliminated on boiling with alcohol, and which in boiling water rapidly affords the stable yellow *N*-acetyl-2 : 3-hydroxynaphthoic anilide, m.p. 152°, previously prepared from acetyl-2 : 3-hydroxynaphthoic acid and regarded as possessing an *O*-acetyl

structure. The brick-red isomeride does not couple with diazo compounds, and its transformation into the *N*-isomeride can be accurately followed by the increment of coupling power. Reasons are advanced for attributing the enol and keto-structures to the red and yellow isomerides, respectively. Treatment of the *O*-acetyl compound with excess of pyridine affords a bright yellow *substance*, m.p. 237°, formed by addition of 1 mol. of pyridine. The reaction appears to be general, other *O*-acetyl derivatives of 2 : 3-hydroxynaphthoic arylides affording similar products with pyridine, pyrrole, or quinoline, etc. In acid media formaldehyde can form a stable α-methylene condensation product which is incapable of coupling, and is the cause of the permanent " fade " which results on prolonged exposure of formaldehyde-protected hanks to atmospheric carbon dioxide etc. R. BRIGHTMAN.

PATENTS.

Manufacture of textile fibres, yarns, fabric, or the like, for obtaining effects of colour or lustre or both. N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (E.P. 249,538, 17.3.26. Conv., 17.3.25. Addn. to E.P. 245,407 ; B., 1926, 820).—The sulphur content of the material (viscose silk) is locally varied by the precipitation of different amounts of sulphur on different parts of the fibre, by local removal of sulphur from uniformly sulphured fibre, or by the action of a desulphurising agent (*e.g.*, 1% sodium sulphide solution) which removes sulphur either from the surface or (after fixation of the surface sulphur by dyeing) from the core of the fibre only. The lustre of the material is regulated by its sulphur content, different colour effects being produced by dyeing the treated material with an agent which combines with or fixes sulphur. B. P. RIDGE.

Manufacture of dye preparations [for acetate silk]. SOC. CHEM. IND. IN BASLE (GES. FÜR CHEM. IND. IN BASEL) (E.P. 249,549, 18.3.26. Conv., 20.3.25).—Dye preparations which readily disperse in water, especially in presence of soap or a sulphonated soap, and are thus suitable for dyeing acetate silk, are made by grinding a non-sulphonated dye, having affinity for acetate silk, with sulphite-cellulose pitch (liguinsulphonic acid) and evaporating the dispersion so obtained, or by merely mixing or grinding together the dry components. C. HOLLINS.

Manufacture of vat dyestuff preparations. SOC. CHEM. IND. IN BASLE (E.P. 250,251, 31.3.26. Conv., 2.4.25). —The vat dye is mixed first with a non-caustic alkali (alkali carbonate, borate, or phosphate) with or without a dispersing agent (Turkey-red oil, sulphite-cellulose lye, etc.) dried, and mixed with sodium hyposulphite to give a dry powder which dissolves in water to give a feebly caustic vat suitable for wool dyeing. Examples are quinone vat dyes and indigoids. C. HOLLINS.

Dyeing and printing animal and vegetable materials. CHEM. FABR. MILCH A.-G., and K. LINDNER (E.P. 246,507, 23.1.26. Conv., 23.1.25).—Sulphonated condensation products of benzene hydrocarbons with aliphatic acid chlorides of not less than 10 carbon atoms are added either to the dye-bath or to the dye-paste to soften the goods to be dyed, to disperse the dye, and to assist penetration. Palmitobenzenesulphonic and stearotoluenesulphonic acids and their salts are effective in small proportions (0·01—0·1% on weight of material to be dyed, 0·1—1% in printing pastes). The compounds are not precipitated by acids, and the effects are increased by addition of a mono- or poly-hydric alcohol. C. HOLLINS.

Dyeing or printing animal and vegetable fibres with mordant dyes. CHEM. FABR. MILCH A.-G., and K. LINDNER (E.P. 269,917, 23.1.26. Conv., 23.1.25).—The material is mordanted with a metal salt (chromium, aluminium, copper, iron, tin, etc.) of (i) a sulphonated condensation product of a benzene hydrocarbon with an aliphatic acid chloride of not less than 10 carbon atoms, or (ii) a polynuclear sulphonic acid containing an *iso*propyl or longer aliphatic side-chain. The sulphonate may also be mixed with the usual mordanting salt. Palmitobenzenesulphonic, stearotoluenesulphonic, and *iso*propylnaphthalenesulphonic acids are specially suitable ; the two first mentioned are prepared from the appropriate condensation product by sulphonation with 7% oleum. The effects are enhanced by addition of mono- or poly-hydric alcohols to the mordanting bath. C. HOLLINS.

Dyeing, printing, and stencilling of products consisting of or containing cellulose esters or ethers. H. DREYFUS (E.P. 272,982, 16.3.26).—Cellulose esters and ethers other than cellulose acetate (cf. E.P. 270,987 ; B., 1927, 553) weighted with salts of tin, zinc, tungsten, or aluminium precipitated on the goods as phosphate, silicate, tannate, etc., have affinity for direct, basic, acid, and other dyes. C. HOLLINS.

Dyeing, printing, and stencilling preparations and dyeing etc. of cellulose acetate materials therewith. BRITISH CELANESE, LTD., and G. H. ELLIS (E.P. 273,819, 10.4.26).—Substantially insoluble dyes, or, in the case of developing or ice colours, dye components, for dyeing, printing, or stencilling cellulose acetate materials are solubilised by means of sulpho-aromatic ricinoleic acids (or their salts) obtained, *e.g.*, by condensing benzene, phenol, naphthalene, etc. with ricinoleic acid or castor oil in 100% sulphuric acid. C. HOLLINS.

Dyeing, printing, or stencilling cellulose acetate materials. BRITISH CELANESE, LTD., and G. H. ELLIS (E.P. 273,820, 10.4.26. Addn. to E.P. 219,349 ; B., 1924, 906).—Resin soaps (sodium resinate) are used in place of the sulphoricinoleic acid etc. of the prior patent as solubilising agents for substantially insoluble dyes having affinity for cellulose acetate. C. HOLLINS.

Reserves for batik dyeing. W. ROESSINGH (Dutch P. 15,039, 23.6.24).—The cloth is treated with a 10% solution of " Algin " or " Norgin " (products obtained from sea algæ), and then passed through a 10% solution of a copper, lead, zinc, or aluminium salt, whereby a layer of the water-insoluble Algin or Norgin salt is precipitated on the material. The fabric is then " cracked " and dyed, the characteristic marbled batik effect being produced. The reserve may be removed by boiling with 10% sodium carbonate solution. C. HOLLINS.

Improving the fastness to rubbing of developed dyeings. CHEM. FARB. STOCKHAUSEN & CIE. (G.P. 438,325, 1.10.24).—In place of Turkey-red oil, ricin-

oleate, or other assistants, there is added to the padding bath a solution of resin in xylene, amyl acetate, acetone, pyridine, benzene, alcohol, hydrogenated naphthalenes, *cyclo*hexanol, etc. The bath remains quite clear even after keeping and when made up with hard water.

C. Hollins.

Dyeing machine. F. Davis (E.P. 273,207, 19.2.27).

Dye jigs for dyeing fabrics. D. McCaig and R. Livingstone (E.P. 273,418, 13.4.26).

Diazo preparations (E.P. 246,181, 246,870, 269,212, 273,352; G.P. 438,743).—See IV.

Starch (E.P. 273,481).—See XVII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Concentration of sulphuric and nitric acids by the Strzoda system. W. Strzoda (Chem.-Ztg., 1927, **51**, 525—526).—In the Strzoda system of concentrating sulphuric acid, the acid is first concentrated to *d* 1·7 in open lead pans and then passed downwards through a series of vertical pipes heated externally by means of producer gas. The vapours evolved in the pipes pass through a tower filled with a suitable filling material, in which they are separated into acid of *d* 1·7 and steam. The former passes back into the concentrating pipes and the latter is condensed in running water in another tower, any nitrous gases present being collected and passed back to the lead chamber. The acid obtained in the process is of 97—98% strength and the loss in the condensed water is only about 50 kg. per 24 hrs. in an 8-pipe system making 6—7 tons of acid per day. A similar plant with additional tubes may be used for concentrating nitric acid by distillation with sulphuric acid followed by re-concentration of the sulphuric acid in another part of the same apparatus.

A. R. Powell.

Depressing action of rhodium and of iridium on platinum in the catalysis of sulphur trioxide. G. R. Levi and M. Faldini (Giorn. Chim. Ind. Appl., 1927, **9**, 223—225).—Probably owing to the ability of rhodium and iridium to form moderately stable oxides, especially sesquioxides, the presence of 10% of either of these metals in the platinum used in the catalytic preparation of sulphur trioxide from dioxide and air causes a marked diminution in the yield; osmium produces a similar but less pronounced effect, whereas palladium or ruthenium in the same proportion increase the yields.

T. H. Pope.

Nitric acid from ammonia. C. L. Parsons (Ind. Eng. Chem., 1927, **19**, 789—794).—Economic changes and technical improvements have during recent years favoured ammonia as a source of nitric acid. The latter include the use of circular gauzes and preheaters with a higher working temperature, and the use of chrome-steel for nitric acid towers, pipe-lines, etc. The largest convenient converter unit has a capacity of 5 tons of nitric acid per 24 hrs. In American practice a gauze of 0·076 mm. wire is used with an estimated working temperature of 1025°. The high temperature reduces the effect of catalyst poisons. A conversion efficiency of 90—97% is obtainable. Chrome-steel (17% Cr) is quite unaffected by nitric acid. Towers of this material are best placed in the open air, and the effective cooling assists absorption. The tower efficiency should be 96%.

C. Irwin.

Sulphate of ammonia plant. F. A. Ernst and W. L. Edwards (Ind. Eng. Chem., 1927, **19**, 768—773).—In the indirect or "hot" method of manufacture of ammonium sulphate, the apparatus can be used without alteration for making ammonium phosphate. The apparatus described has a small brick-packed absorption tower, through which the acid flows into the saturator, and a rotary drier for the centrifuged salt. A saturator temperature of 100° and an acidity of 7% is recommended. Theoretically, 164 gals. of water are evaporated per ton of sulphate made, whence the admissible quantity of wash water can be calculated. A speed of 350 r.p.m. should not be exceeded on the centrifugal, leaving 3% of moisture in the salt. Constructional details for a plant to make 60 tons of sulphate per 24 hrs. are given.

C. Irwin.

Manufacture of ammonium chloride [from gas liquor]. W. Adolphi (Chem.-Ztg., 1927, **51**, 489—491).—When ammonium sulphate is treated with an alkali chloride some sodium sulphate remains in the ammonium chloride; consequently, strong gas liquor is usually treated with hydrochloric acid. The gas liquor must be free from oil and tar, whilst the content of carbon dioxide, sulphur, and thiocyanates should be low. The acid (*d* 1·13—1·15) must not contain more than 1% of sulphuric acid because the ammonium sulphate formed will decompose to sulphite at the sublimation temperature of 250°, but the iron content is not important. Saturation takes place in a stone vat connected with a flue to lead off hydrogen sulphide, the acid and liquor being added in a regular stream and agitated with compressed air; the process is stopped while the solution is slightly acid, to agglomerate the sulphur. The nearly saturated solution is passed through a filter press with wooden frames, ammonia being recovered periodically from the precipitate, and the solution is concentrated to about 500 g./litre of ammonium chloride in a lead vessel fitted with lead steam coils and is run off by gravity to lead crystallising vessels. After 48 hrs., the contents set in a crystalline mass with relatively little mother-liquor; these are subsequently drained, dried by gentle heat, and sublimed. The sublimation vessel is hemispherical, and made of cast iron lined with a double layer of special glazed bricks cemented with alumina and alkali. The cover is asbestos-coated cast iron, and has an opening for the escape of moisture and non-condensed ammonium chloride vapour, which is recovered in a tower. A seal is made between the vessel and the cover with dry sand. The charge is fired strongly for 2—3 days, then gently and uniformly for 10—12 days, when the sublimed salt is detached from the cover. An 84—88% yield is obtained, the losses being due to volatility of the ammonia, the sulphuric acid in the hydrochloric acid, and to mechanical and sublimation losses.

W. G. Carey.

Separation and determination of mixtures of iodide, bromide, and chloride. O. Chick (J.S.C.I., 1927, **46**, 327—329 T).—On titrating a solution acidified

with nitric acid containing a mixture of iodide, bromide, and chloride with $0 \cdot 1N$-silver nitrate solution, the iodide is precipitated first, the iodide end-point being determined by a drop of the solution failing to give a blue colour with starch–nitrite paper. The bromide is precipitated next, the combination being complete when the solution, which has been rendered alkaline with ammonia, completely clarifies on thorough shaking, and when the precipitate given by the silver solution completely redissolves. The chloride is precipitated last, and is determined by Volhard's method after re-acidifying the mixture with nitric acid.

Decomposition of crude [calcium] phosphate with sulphurous acid and with ammonium sulphite. W. Stollenwerk (Z. angew. Chem., 1927, **40**, 553—559).—Treatment of insoluble calcium phosphate with sulphurous acid in sixfold excess transforms it almost completely into soluble calcium monophosphate. No free phosphoric acid is thus obtained. Ammonium sulphite is considerably less effective in bringing about the formation of soluble phosphate. L. M. Clark.

Production of titanium tetrachloride. K. F. Beloglazov (Contrib. Study Nat. Resources U.S.S.R. [Russia], 1926, No. 56, 20—23).—Briquettes prepared from titanium ore (ilmenite) ground to 0·5 mm., carbon, and starch paste are first heated in the absence of oxygen and then chlorinated, the residue being used in the next charge. Chemical Abstracts.

Obtaining titanium tetrachloride from titanium carbide. S. I. Oreshkin (Contrib. Study Nat. Resources U.S.S.R. [Russia], 1926, No. 56, 14—20).—Titanium carbide and chlorine at 200° yield titanium tetrachloride; titanium dioxide, carbon, and chlorine react at a higher temperature and more slowly. When there is 20—30% of carbide in the electric furnace, ferric chloride does not hinder the process, being removed by the titanium tetrachloride. Chemical Abstracts.

Titanous chloride for the determination of iron and of chloric acid. P. S. Brallier (Ind. Eng. Chem., 1927, **19**, 846—848).—A simplified procedure for the determination of iron and of chloric acid by means of titanous chloride solution, suitable for use in small industrial laboratories, consists in adding a known excess of titanous chloride solution to the solution under examination in presence of hydrochloric acid and titrating the excess with standard ferric ammonium sulphate solution; a blank determination is performed with the titanous chloride solution, which need not therefore be kept at constant concentration. The method is convenient for the determination of chlorates in sodium hydroxide solutions from electrolytic cells and evaporators, and in hypochlorite solutions. W. J. Powell.

Stability of titanium sulphate solutions. H. Wilkinson and A. G. Tyler (J. Soc. Dyers and Col., 1927, **43**, 225).—Protection of the stock bottle of titanium sulphate solution from atmospheric oxygen with absorption tubes containing alkaline pyrogallol solution enables the solution to be used for accurate analytical work (cf. B., 1927, 364). R. Brightman.

Treatment of sulphur ores in cell furnaces. M. Gatto (Giorn. Chim. Ind. Appl., 1927, **9**, 225—227).—Polemical. T. H. Pope.

Development of the synthetic nitrogen industry in Great Britain. G. P. Pollitt (J.S.C.I., 1927, **46**, 291—295 t).

Ammonium thiocyanate. Gluud and Klempt.—See II.

Solubility of basic slag. Dunkel.—See X.

Lime in sugar industry. Kukharenko.—See XVII.

Patents.

Manufacture of nitric acid. E. L. Nellis, Assr. to Trojan Powder Co. (U.S.P. 1,635,949, 12.7.27. Appl., 29.10.21).—When distilling nitric acid from sodium nitrate and sulphuric acid, about one quarter of the total sulphuric acid used in a distillation is reserved and towards the end of the process is passed into the still in counter-current to the weak distillate. W. G. Carey.

Fixation of atmospheric nitrogen. O. Ficker (F.P. 612,477, 8.3.26. Conv., 15.2.26).—Solutions containing ferrous salts, rendered weakly alkaline and inoculated with nitrogen-fixing soil bacteria, rapidly absorb atmospheric nitrogen at the ordinary or at raised temperatures, until the iron is completely oxidised. L. A. Coles.

Production of nitrates from calcium cyanamide. K. A. Hofmann (G.P. 439,510, 14.10.25).—Calcium cyanamide alone or in admixture with alkaline compounds is treated with oxidising gases at a temperature below the decomposition point of the desired nitrate, preferably in the presence of catalysts such as copper, nickel, silver, or oxides of copper, bismuth, or nickel. L. A. Coles.

Separation of water from mixtures of ammonia and water vapour. C. Cooper, D. M. Henshaw, and W. C. Holmes & Co. (E.P. 272,970, 16.12.25).—Vapours from an ammonia still pass through a distillation column in counter-current to a solution of potassium carbonate or other material having a selective affinity for water, the liquid being heated at 90—100°. The spent liquid is reconcentrated for re-use by causing it to flow over the outside of the column. W. G. Carey.

Manufacture of ammonium chloride crystals. J. W. Moore, W. G. Polack, and Castner-Kellner Alkali Co., Ltd. (E.P. 273,093, 25.6.26).—Anhydrous ammonia and gaseous hydrochloric acid, preferably both synthetic and containing a controlled proportion of hydrogen, are caused to react at such velocity and/or with such thermal insulation that the reaction temperature is considerable. Part or all of the ammonium chloride produced is in tough fibrous crystals, similar to those produced by sublimation with external heat. W. G. Carey.

Manufacture of alkali bisulphate. I. G. Farbenind. A.-G. (E.P. 268,817, 1.4.27. Conv., 1.4.26).—The manufacture of alkali bisulphate from chlorides and sulphuric acid is conducted in an electrically-heated rotary drum of acid-resisting cast iron or ferrosilicon, which is itself the heating resistance or is heated by a surrounding sheet metal cylinder insulated from the drum, current being conducted thereto by slip rings and brushes. W. G. Carey.

Production of solid ammonium carbonate from its components. Rhenania-Kunheim Verein Chem.

Fabr. A.-G. (E.P. 262,408, 11.11.26. Conv., 7.12.25).—Gaseous ammonia and carbon dioxide are introduced into a reaction vessel, the walls of which are moistened continuously or intermittently with water, ammonium carbonate solution, or ammonia liquor, the liquid dripping from the reaction vessel being repeatedly used for this purpose. W. G. Carey.

Manufacture of ferric sulphate. B. Hart, Harris, Hart & Co., and Refiners Ltd. (E.P. 273,883, 12.6.26).—Ferric sulphate, suitable for use as an active material for the purification of oils, spirits, and fats, is made by dissolving ferrous and/or ferric oxides and/or hydroxide (*e.g.*, waste oxidised iron borings from aniline manufacture) in sulphuric acid and adding further sulphuric acid together with an oxidising agent to precipitate ferric sulphate. W. G. Carey.

Production of barium peroxide. J. P. Poët (F.P. 615,381, 2.4.26).—Barium carbonate is converted into barium peroxide in a furnace system containing two or more thin-walled galleries for the barium carbonate, an intensive circulation of combustion gases serving for heating the furnace. L. A. Coles.

Refining and separation of [oxysalts of] metals [arsenic and tin]. H. Harris (E.P. 273,440, 7.5.26. Cf. E.P. 245,479; B., 1926, 236).—Sodium oxysalts of tin and arsenic are separated by treatment with a cold solution of caustic soda of such strength that the tin salt is dissolved without the arsenic (or any antimony present) being attacked. After filtration the tin in the sodium stannate solution is recovered electrolytically and the caustic soda used again, sodium arsenate being recovered from the insoluble portion. The strength of the caustic soda solution is 100—250 g./litre depending on the extent of separation required and the amount of caustic soda or salts already present. W. G. Carey.

Decomposition of aluminous material. Büttner-Werke A.-G., and F. Kleinmann (G.P. 439,540, 14.1.23).—The unroasted material is ground wet before or during the usual decomposition with alkali hydroxides, and during the grinding and decomposition processes the material is treated with oxidising agents which do not hinder the decomposition process and the subsequent precipitation of aluminium hydroxide, such as chlorine, chlorates, peroxides, or hypochlorites. L. A. Coles.

Treatment of clays and the like for production of new compositions. J. G. Cloke (E.P. 272,976, 17.2.26).—Argillaceous material is treated, preferably while boiling, with a reagent, *e.g.*, a strong mineral acid, which dissolves out the staining material without decomposing the clay. The treated material is then subjected to the action of fluorine or a fluorine compound, such as nascent hydrofluoric acid, which precipitates the clay as a product closely resembling mica when washed and dried. B. W. Clarke.

Obtaining sulphur from alkaline-earth sulphates. Salzwerk Heilbronn A.-G., T. Lichtenberger, and K. Flor (E.P. 273,841, 16.4.26. Addn. to E.P. 251,942; B., 1927, 75).—Instead of dissolving the crude sulphate in molten sodium chloride, other alkali or alkaline-earth salts are used, provided they are fusible without liberation of water and do not decompose the fused sulphates; alkali sulphates or alkali and alkaline-earth fluorides are suitable. If salts are employed which yield acids when steam is blown in after the reduction, hydrogen sulphide is liberated and an alkali or alkaline-earth salt regenerated. W. G. Carey.

Manufacture of active adsorbent and catalytic masses. I. G. Farbenind. A.-G. (E.P. 255,904, 26.7.26. Conv., 27.7.25).—An irreversible colloid, *e.g.*, silica, alumina, iron oxide, is prepared as a sol or jelly, and, before being dried, is mixed intimately by mechanical means with one or more catalytic substances in the liquid or finely-divided solid state. When the colloid is a sol the mixture is allowed to set to a jelly, dried, and purified by washing before or after complete drying. The catalytic substances or their decomposition products may be removed by acids after drying completely or partially, preferably after saturation with water vapour. W. G. Carey.

Metal vessels for use in the production and storage of hydrogen peroxide. G. Schmidt (G.P. 439,834, 14.10.21).—Vessels constructed of chromium steels such as Krupp's V2A and V1M steel, are suitable for the production and storage of hydrogen peroxide and other solutions containing active oxygen, or for processes involving the use of such solutions, even at raised temperatures. L. A. Coles.

Treating gases with liquids (E.P. 273,103).—See II.

Washing and bleaching powders (E.P. 273,414).—See XII.

VIII.—GLASS; CERAMICS.

Recuperative glass furnaces. T. Teisen (J. Soc. Glass Tech., 1927, **11**, 265—279).—The latest form of the author's recuperative pot furnace, which includes a new hexagonal type of recuperator tube, is described. A. Cousen.

Function of arsenic in soda-lime-silica glass. II. E. M. Firth, F. W. Hodkin, and W. E. S. Turner. **III. Effect of temperature and time.** E. M. Firth, F. W. Hodkin, W. E. S. Turner, and F. Winks (J. Soc. Glass Tech., 1927, **11**, 190—205, 205—214. Cf. B., 1926, 585).—II. In a series of commercial soda-lime glasses made in tank furnaces the arsenic retained in the glass ranged from 55% to over 90%, and, in general, a large proportion had been converted into the quinquevalent state. Neither nitre nor carbon has much effect at 1300—1400° on eliminating arsenic from glass unless the amount of arsenic used is considerable and the nitre or carbon much in excess of the arsenious oxide. The main function of these agents appears to be oxidation or reduction of the arsenic, respectively. The combination of nitre and arsenious oxide increases the rate of melting as compared with that of batches containing arsenious oxide only, and whilst increasing addition of carbon with the arsenic decreases the rate of melting it increases the fining rate. The results do not confirm the theory that the refining action is due to progressive dissociation of arsenic oxide. Little effect on fining is found in plunging lumps of arsenious oxide into the glass after melting, although, under certain conditions, a commercial method is stated to give more rapid fining by this means. Little difference of working

properties is found in glass made from batches containing both arsenious oxide and nitre, but the latter gives an additional decolorising effect. A good soda-lime glass containing 0·27% of arsenious oxide showed no loss of the oxide by extraction in 6 hr. boiling tests with vinegar and concentrated sugar solution, and the amount extracted under similar conditions by 2*N*-sodium carbonate corresponded to only 0·2% of the total loss of weight.

III. At 1500° soda-lime batches containing arsenious oxide melt more rapidly than at 1300—1400°, but the rate of fining is not uniformly increased with increased temperature. Glasses so melted are generally heterogeneous, due largely to pot attack and not to the presence of the arsenic. The proportion of arsenic remaining in the glass is greatest with lowest concentrations in the batch, the average amount retained being over 70%. With a founding temperature of 1400° a glass containing 0·75% of arsenious oxide loses no arsenic over a period from 9 to 44 hrs. from the start of founding, but at 1500° the loss is progressive.

A. Cousen.

Decomposition of glass by water at high temperatures and pressures. G. W. Morey and N. L. Bowen (J. Soc. Glass Tech., 1927, 11, 97—106).—Glass in the form of 1 cm. cubes or tubing was heated with water in steel bombs for about 20 hrs. at 300° or 550°, and the product obtained microscopically examined. With crown and borosilicate-crown glasses quartz crystals and an unknown crystalline phase appeared. Barium crowns gave mostly crystals of barium silicate ($BaO,2SiO_2$). Flint glasses with low lead oxide content were badly decomposed at 300° and practically completely so at 550°, giving a hydrous glass and quartz crystals. Increase of lead content gave increased stability, dense flints showing great resistance. With a content of 60% of lead oxide lead metasilicate crystallised. In each of the optical types there was correspondence between the amount of attack and the iodoeosin number. Of a number of commercial glasses examined, Jena combustion was least attacked, whilst the failure of pyrex was outstanding.

A. Cousen.

Analysis of the simpler glasses. W. H. Withey (J. Soc. Glass Tech., 1927, 11, 124—153).—The methods usually employed for the analysis of silicate glasses of the more common types were critically examined, a scheme of procedure being drawn up. Glasses comprised in the scheme were those containing silica and the oxides of iron, aluminium, manganese, calcium or lead, magnesium, sodium, and potassium, with sulphates and chlorides, and sometimes the oxides of arsenic and antimony.

A. Cousen.

Analysis of glasses. V. Dimbleby (J. Soc. Glass Tech., 1927, 11, 153—166).—After outlining the chief precautions to be taken in the analysis of simple alkali–lime–magnesia–silica and alkali–lead oxide–silica glasses the following special determinations in glasses are described :—Iron oxide, manganese oxide, arsenious oxide, titania, zirconia, zinc oxide, cobalt oxide, barium oxide, alumina, boric oxide, sulphur trioxide, and fluorine.

A. Cousen.

Behaviour of fluorides in glasses and enamels. II. G. Agde, H. F. Krause, and W. M. Lehmann (Z. angew. Chem., 1927, 40, 804—808).—Seven members of a series of fluoride-opal glasses, previously microscopically examined (cf. B., 1927, 483), were subjected to *X*-ray analysis. In the cases in which sodium fluoride or aluminium fluoride had been used as opaliser, the precipitate was found to be sodium fluoride, whilst a glass opalised by calcium fluoride contained a precipitate of calcium and sodium fluorides in varying porportions. The results confirmed the microscopical tests.

A. Cousen.

Selective absorption of ions by colloidal clay. A. Demolon and G. Barbier (Compt. rend., 1927, 185, 149—150).—The absorbability of cations by clay appears to be constant and independent of the nature of the ions fixed. When mixtures of salts are used, a phenomenon of distribution is observed, the ions K and NH_4 being fixed in sensibly equivalent proportions, whilst the ion Na is only feebly absorbed in the presence of the other two ions. The ion Ca of calcium chloride diminishes, to an extent depending on its concentration, the fixation of the alkaline ions. By varying the concentrations of these four ions, it is possible to modify at will the magnitudes of their respective absorptions and thus effect selective absorption without a membrane. Similar results are obtained when soil is used in place of the clay.

T. H. Pope.

Analysis of refractories. W. J. Rees (J. Soc. Glass Tech., 1927, 11, 172—176).—Methods applicable to the analysis of glass could generally be applied to the analysis of refractory materials, but extremely fine grinding before fusion was necessary. Short methods are given for the analysis of silica brick, dolomite, chrome refractories, and sillimanite refractories.

A. Cousen.

Analysis of silicate slags. T. P. Colclough (J. Soc. Glass Tech., 1927, 11, 177—184).—A description of the main points of the analysis of slags in the iron and steel industry, in which the volumetric determination of lime is strongly recommended. Rapid control methods for blast furnace, acid or basic open-hearth, hot metal mixer, and electric furnace slags are dealt with.

A. Cousen.

Alloys for moulding glassware. Bauret, Portevin, and Chevenard.—See X.

Patents.

Frosting glass. Siemens & English Electric Lamp Co., Ltd., P. D. Oakley, and J. N. Aldington (E.P. 273,046, 16.4.26).—Glass surfaces, especially electric lamp bulbs, are frosted by a single solution containing an alkaline fluoride or fluorine-containing substance together with a suitable amount of hydrochloric acid and/or soluble chloride, whereby a cellular structure of the etched surface results. If, in addition, sulphuric acid or a soluble sulphate is added, the opacity is increased without loss of cellular structure. The time of exposure depends on the composition of the glass, and the temperature must be carefully controlled. The uniformity of the result is increased by addition of a suitable proportion of water-glass.

A. Cousen.

Manufacture of silica articles with glazed surface, more particularly for laboratory use. Quartz & Silice (E.P. 273,636, 7.6.27. Conv., 15.7.26).—

Crucibles, capsules, etc. are prepared from previously moulded blanks of a shape approximating to that desired, but of rather larger dimensions, by remelting these so that they contract under the action of gravity and of surface tension and assume the contours of a hollow, water-cooled pattern of nickel or suitable nickel alloy. Heating is most conveniently performed by a high-frequency induction furnace with graphite muffle. The process ensures exact dimensions, regular and smooth surfaces, strength, and homogeneity.

A. COUSEN.

Decolorising china clay and the like. P. E. WICKHAM (E.P. 273,373, 23.3.26).—Clay made into a paste with water is treated with a solid hyposulphite and an acid, preferably organic (*e.g.*, formic acid), to remove organic colouring matter. The clay is then collected on a filter press and washed.

B. W. CLARKE.

Machine for sifting potter's slip, powdered substances, etc. F. BEARDMORE, and W. BOULTON, LTD. (E.P. 273,387, 30.3.26).

IX.—BUILDING MATERIALS.

Permanganate value of clays. H. TER MEULEN (Chem. Weekblad, 1927, **24**, 351—352).—The suitability of clay for brickmaking is thought to depend to some extent on its content of humus, which may be determined, in the absence of ferrous compounds, nitrites, and chlorides, by the permanganate value. The sample is boiled in presence of sulphuric acid, with a known quantity of permanganate, a known excess of ammonium oxalate is then added, and the excess of oxalic acid titrated with standard permanganate. S. I. LEVY.

Effects of calcium chloride on road slab concrete. H. P. OLSON (Eng. News-Rec., 1927, **99**, 69).—The admixture of 1% and 2% of calcium chloride with the concrete shows, as the result of beam and cylinder tests, no appreciable gain in strength at 7 days, whilst at 28 days the strength is less than that of plain concrete. Surface treatment of the slabs with calcium chloride solution shows a big increase in strength compared with concrete containing an admixture of the chloride. A crack survey of the pavement shows that the surface-cured slabs are comparatively free from cracks.

B. W. CLARKE.

PATENTS.

Manufacture of a lime sludge product. C. J. HERRLY, Assr. to PREST-O-LITE Co., INC. (U.S.P. 1,635,212, 12.7.27. Appl., 19.2.24).—Lime sludge is mixed with a cellulosic material and an inorganic, water-absorbing material to form a plastic composition which is shaped and baked at above 200°, forming building blocks and the like. B. W. CLARKE.

Colouring of cement, concrete and the like. BRITISH DYESTUFFS CORP., J. BADDILEY, A. SHEPHERDSON, and A. DAVIDSON (E.P. 273,477, 19.6.26).—Vat dyes, either already dispersed or in easily dispersible form, are added to the water used in gauging cement, concrete, etc. Thioindigo Red B gives a pink colour, Indanthrene Blue or Bromoindigo 4B a blue, Duranthrene Golden Orange Y a yellow, Duranthrene Claret a bordeaux.

C. HOLLINS.

Enamelling plates or objects of fibrous cement with vitreous enamels. FIBREMO, SOC. COOPÉRATIVE (E.P. 263,195, 18.12.26. Conv., 19.12.25).—Fibrous cement articles, dried and degreased in a muffle furnace, are coated with a mixture of an enamel and a flux, which is vitrified by firing at a suitable temperature. The flux consists preferably of a fused mixture of silex, red lead, borax, and potassium nitrate, disintegrated by pouring into cold water. B. W. CLARKE.

Bituminous emulsions for use in making and surfacing roads and the like. UNIVERSAL RUBBER PAVIORS (MANCHESTER, 1923), LTD., and S. J. PEACHEY (E.P. 273,031, 6.4.26).—A suitable proportion (about 8% by wt.) of gutta-percha pitch or balata pitch is incorporated by violent agitation with a bituminous emulsion at about 100°. B. W. CLARKE.

Preparation of a composition for preserving wood, timber, etc. G. GUNN (E.P. 273,007, 26.3.26) —Timber is impregnated with an aqueous solution of a dichromate together with a solution of a copper salt acidified with acetic acid preferably in the form of pyroligneous acid. B. W. CLARKE.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Cause of temper-brittleness in steels. K. HONDA and R. YAMADA (Sci. Rep. Tôhoku Imp. Univ., 1927, **16**, 307—319).—The phenomenon of temper-brittleness in steels is discussed, and is attributed to the separation of carbides along the grain boundaries during slow cooling (cf. Andrew and Dickie, B., 1926, 826). Electrical resistance measurements indicate that in manganese or nickel–chromium steels the solubility of the carbides in ferrite is practically constant below 400° but increases above this temperature, and hence on slow cooling from a tempering temperature the carbides precipitate on the boundaries of the sorbitic grains. Temper-brittleness is observed by both impact and statical bending tests. The necessary condition for the phenomenon to be well marked is the notch in the test bar, which creates a large stress intensity so that the fracture starts from the grain boundaries. The effect of temperature on the impact resistance of ordinary non-susceptible and suceptible steels is discussed. The minimum impact resistance in the region 550—600° ("blue shortness") is due to the effect of strain-hardening during the impact, superimposed on the temperature–plasticity relations. W. HUME-ROTHERY.

Faults occurring during the working of mild steel and means of avoiding them. F. KÖRBER (Stahl u. Eisen, 1927, **47**, 1157—1166).—Overheating of mild steel articles, *e.g.*, during welding, causes a rapid increase in grain growth and a corresponding decrease in resistance to impact. A similar coarse and weak structure is obtained by annealing critically-rolled steel within a certain temperature range between 650° and 900°, but the structure may be refined again by heating at 900—950°, *i.e.*, just above the A3 point. Some varieties of mild steel undergo ageing changes after cold-rolling, resulting in a considerable decrease in tensile strength and resistance to corrosion; if this type of steel

is subsequently heated at 200—400°, *e.g.*, during lacquering, it becomes so brittle that a slight knock suffices to cause fracture of thin articles. The addition of 4—5% of nickel to the steel required to be cold-worked and subsequently heated at 200—400°, prevents the development of any weakness, or, alternatively, the cold-worked metal should be heated for 15 min. at 500°. Examples of the effect of blowholes and mechanical inclusions in the steel on the working properties are given.

A. R. POWELL.

Solubility in citric acid of the phosphoric acid of basic slag. T. DUNKEL (Stahl u. Eisen, 1927, **47**, 1205—1207).—The percentage of phosphoric acid in basic slag that is soluble in citric acid is independent of the rate of cooling and depends solely on the ratio $SiO_2 : P_2O_5$. When this ratio exceeds 0·315 practically the whole of the phosphoric acid is soluble. The slag at this point has the composition $4Ca_3P_2O_8,3Ca_3SiO_5$. On the basis of these results the necessary silica to be added to the converter charge for pig-iron of different silicon contents has been determined, the results being reproduced in graphical form. A. R. POWELL.

Alloys to resist the effects of an alternating temperature-gradient, and, in particular, alloys for moulding fine glassware. P. BAURET, A. PORTEVIN, and P. CHEVENARD (Compt. rend., 1927, **184**, 1655—1656).—Forged ferro-nickel alloys containing chromium and tungsten have been found most suitable for metallic moulds for use with molten or pasty materials. At 20° their dilatability is 8×10^{-6}, and their coefficient of thermal conductivity 0·02 c.g.s. unit; their tenacity is 65 kg./cm.^{2} and the stretching at the point of rupture is 30%. They have a low rate of viscous extensibility and have been used successfully for over two million operations. J. GRANT.

Determination of tungsten in ferro-tungsten and tungsten steels. L. MOSER and K. SCHMIDT (Z. angew. Chem., 1927, **40**, 667—668).—The sample is powdered, placed in a porcelain boat, and heated in a tube in a furnace in a current of air and carbon tetrachloride vapour. The mixture of tungsten and iron chlorides is received in nitric acid, the solution evaporated to dryness, and the tungsten oxide obtained freed from iron by evaporating to fuming twice with a mixture of equal parts of ammonium chloride and ammonium bromide. If potassium chloride be added to the sample, and the treatment effected below red heat, the iron chloride remains in the boat as the non-volatile double potassium chloride. S. I. LEVY.

Assay of rolled-gold plate. R. GILCHRIST (Ind. Eng. Chem., 1927, **19**, 827—829).—"Rolled gold" articles consist of a base metal core and gold alloy surface, the latter up to 0·076 mm. thick, and are drawn from billets of the two alloys brazed together. In analysis, the base metal is usually dissolved by a stripping solution. This leaves a loose black surface at the junction where there is a zone of alloy of varying composition. A series of tests were made and the results compared with those given by samples of the gold alloy removed by milling. Nitric acid solutions were found to give a slightly high result for the fineness of the alloy, and ammoniacal solutions one slightly low. Whether the black surface of the treated specimen was rubbed off or not made no appreciable difference. C. IRWIN.

Anodic formation of powdered copper and passivity of the anodes. E. DENINA (Annali Chim. Appl., 1927, **17**, 284—296).—Thompson's explanation of this phenomenon is based on the inaccurate assumption that the whole of the copper of the electrolyte exists in the ionic form (cf. B., 1926, 588). The author's discussion of the mechanism of anodic dissolution leads to the hypothesis that the ratio of the amount of cuprous to that of cupric ions formed diminishes as the current density increases; this would explain the beneficial effect of augmenting the current density. T. H. POPE.

Influence of the concentration of [nitric] acid on the acid consumption in the yellowing of brass. E. DORN (Z. Metallk., 1927, **19**, 280—281).—In pickling brass with nitric acid alone to obtain a yellow finish, the pickling bath becomes ineffective when the acidity falls below 30%. By adding one sixth of its volume of sulphuric acid to the bath it can be used until it contains 25% of free nitric acid, and with one third of its volume of sulphuric acid until it contains 10% of free nitric acid. The addition of sulphuric acid to the bath decreases the initial rate of attack on the metal, but increases the velocity of the reaction towards the end. A. R. POWELL.

Behaviour of bronze in cellulose bleach liquors. W. HEIKE and F. WESTERHOLT (Z. Metallk., 1927, **19**, 285—287).—The effect of the tin and lead content and of annealing on the bronze used in a screw conveyor working in a mixture of cellulose and bleach liquor (sodium hypochlorite) has been investigated. With up to 6% Sn the rate of corrosion was accelerated by annealing at 700°, and with more than 8% Sn it was retarded, due to equalisation of the tin distribution by the formation of the homogeneous α from the $\alpha + \beta$ structure of the cast alloy. The alloy with 10% Sn was more resistant after rapid cooling than after slow cooling, but alloys with less tin showed no variation in the rate of corrosion with the rate of cooling. Addition of up to 9% Pb had very little effect on the corrodibility of the alloys either before or after annealing. It therefore appears that the tin content alone has an important bearing on the corrosion of bronze by hypochlorite liquors, a high tin content evenly distributed giving the most satisfactory results. A. R. POWELL.

Acid-resisting bronzes for sulphite-cellulose works. H. RAUCHBERG (Papierfabr., 1927, **25**, 473—477).—After three weeks' immersion in a cellulose boiler using a solution containing 4·65% of free sulphur dioxide, copper lost 7·5% in weight, lead 0·7%, and tin 0·05%, whilst zinc was completely dissolved, and all bronzes containing zinc were more readily attacked than copper. Addition of lead to a bronze increased its resistance to corrosion appreciably, whilst even a very small quantity of antimony improved still further the resistance of a lead bronze. A boiler having the longest life was obtained with alloys within the following range of composition: 15—19% Pb, 5—10% Sn, 1—5% Sb, and 66—75% Cu. The 8% copper–aluminium alloy was very rapidly corroded by sulphurous acid. A. R. POWELL.

Electric smelting of tin ores. F. Freude (Continental Met. Chem. Eng., 1927, **2**, 70—72).—In the electric furnace method the large volume of gas produced is eliminated, and the fused mass is kept saturated with dissolved tin oxide, until the end of the process, the carbon in the upper layers reacting with the tin oxide as it sinks, yielding ferric oxide and tin; 94% of the tin charged is recovered as nearly pure raw tin. The use of the furnace is described; it is also advantageous in the direct treatment of sulphide ores.

Chemical Abstracts.

Pure nickel and technical nickel. F. Mylius (Z. Metallk., 1927, **19**, 261—264).—The composition of various grades of technical nickel is discussed, and details of a method of preparing chemically pure nickel on a laboratory scale are given. Commercial electrolytic nickel may contain up to 1% or more of impurities, chiefly cobalt, zinc, iron, and copper, whilst Mond nickel usually contains 0·2% Fe, 0·05% Cu, and 0·25% C. A sample of the purest Kahlbaum nickel contained 0·02% Co, 0·012% Fe, and traces of other metals. The purest nickel is obtained by the reduction of nickel oxide by hydrogen, and can be worked into rod or sheet without contamination only by a sintering and swaging process resembling that used for working tungsten powder into rod.

A. R. Powell.

Vanadium. J. W. Marden and M. N. Rich (Ind. Eng. Chem., 1927, **19**, 786—788).—Metallic vanadium was prepared by heating the pentoxide with calcium and calcium chloride in a bomb at 950° for 1 hr. $V_2O_5 + 5Ca + 5CaCl_2 = 2V + 5CaO,CaCl_2$. A small piece of sodium is added to remove air and moisture. The treated charge was added slowly to a large volume of cold water with stirring, settled, and the residue washed with dilute hydrochloric acid. The vanadium is left behind as beads. These are soft and ductile, and may be cold-worked into wire. The metal was found to contain over 99% V, with traces of silicon, iron, etc. It had d 6·0, sp. resistance 26×10^{-6} ohm per c.c., and m.p. 1700°. It is rendered brittle by heating with hydrogen and resembles tantalum in its general properties.

C. Irwin.

Properties of pure aluminium. L. Guillet (Compt. rend., 1927, **185**, 163—165).—The properties of "pure" (99·8%) aluminium differ little from the usual commercial aluminiums (99·0—99·5%). The resistivity is a little less, the striction higher, and the total evolution of gas (chiefly hydrogen) is 34·24 c.c. per 100 g. of metal.

J. Grant.

[Aluminium] alloy resistant to sea-water. R. Sterner-Rainer (Z. Metallk., 1927, **19**, 282—284).—The aluminium alloy known in Germany as "KS-Seewasser" contains 3% Mn, 2·5% Mg, and 0·5% Sb; its structure shows finely-divided $MnAl_3$ dispersed evenly throughout a ground-mass of a solid solution of magnesium in aluminium, the antimony not being visible as a separate constituent. The chill-cast alloy has a tensile strength of 18—20 kg./mm.², with an elongation of 3·8%, and a Brinell hardness of 60, whilst for the hard-rolled alloy the corresponding figures are 32 kg./mm.², 4·6%, and 95. After 4 months' immersion in the Baltic Sea at 10° the loss in weight of the cast alloy was 3 g./m.² In contact with bronze the loss was 40 g./m.² and in contact with steel 19/m.² Sand-cast bowls of this alloy have given efficient service in the evaporation of neutral salt solutions.

A. R. Powell.

Rate of dissolution of ultra-light magnesium alloys. A. Portevin and E. Pretet (Compt. rend., 1927, **185**, 125—127).—The rates of corrosion of pure magnesium by $0 \cdot 1N$-hydrochloric and -citric acids have been compared with those of its ultra-light alloys (containing 4—5% Al, Cd, Cu, Ni, Pb, or Zn) by various methods. For this purpose the thermal and gas-evolution methods are preferable to the measurement of the loss in weight. The latter, however, does not give a confirmation of Boguski's law for determining the dissolution constant, when cylindrical samples are used. The conditions necessary for obtaining this constant are outlined. In the thermal method the ratio of the maximum temperature reached during corrosion to the time required to attain it is measured under standard conditions. In general, the rates of corrosion as determined by the two methods follow the same order for a particular reagent, but this order is not the same for the two reagents. Pure magnesium is less attackable than its alloys, but this property is modified by the presence of silicon.

J. Grant.

Cadmium as a plating-metal for utensils. A. Gronover and E. Wohnlich (Z. Unters. Lebensm., 1927, **53**, 392—396).—The deposition of cadmium by electroplating or spraying for the protection of metals (cf. B., 1926, 133, 243, 542), and its use in alloys and solders (cf. B., 1926, 245), are reviewed. The physiological action of the metal and its salts is discussed, and it is shown that a dose of 0·03 g. of a cadmium salt is fatal to animals, whilst very small quantities (not stated) are poisonous to human beings. To test the suitability of the metal for coating cooking utensils, its solubility in acids has been investigated. The plating adheres firmly and resists bending without cracking, but it is readily attacked by acetic and tartaric acids. A surface of 50 sq. cm. exposed to 0·5% acetic acid for 2, 6, and 18 hrs. gave 5·3, 8·0, and 10·8 mg. respectively. Increasing the acid concentration to 2·5% had very little effect on the weight of metal dissolved. The solubility of zinc under the same conditions is appreciably less. To investigate the action of foodstuffs, a sheet of 10 sq. cm. was immersed in plum jam for 3 days, and it was found that 15 mg. of metal per 50 sq. cm. were dissolved. In view of its ease of solubility and its toxicity, the use of cadmium for articles which come into contact with foodstuffs is not recommended.

H. J. Dowden.

Deoxidation of nickel. G. Masing and L. Koch (Z. Metallk., 1927, **19**, 278—279).—See B., 1927, 113.

Analysis of silicate slags. Colclough.—See VIII.

Anode effect in the aluminium bath. Arndt.—See XI.

Patents.

Manufacture of sponge iron. P. W. Nevill (U.S.P. 1,635,950, 12.7.27. Appl., 21.11.25. Conv., 1.12.24).—Iron ore and carbonaceous fuel without flux are passed through a shaft furnace to which air is supplied at several points, so that reduction extends over a

considerable zone and excessive local heating, with consequent fusion, is avoided. W. G. Carey.

Manufacture of steel products. R. A. Hadfield (E.P. 273,855, 29.4.26).—As a step in the manufacture of sound steel ingots a sample of normally finished steel is tested in a small mould which allows of slow cooling, *e.g.*, green sand. If the steel is found to be of a rising nature, a deoxidising agent is added to the charge and the metal corrected before casting the complete heat. C. A. King.

Rust-proofing iron. E. C. R. Marks. From Parker Rust Proof Co. (E.P. 273,168, 16.2.26).—Articles of iron are rendered rust-proof by treating them in a solution of the dihydrogen orthophosphate of any one or more of the metal groups, iron, zinc, manganese, or cadmium, the salts of the metals having been prepared according to E.P. 270,820 (B., 1927, 522). C. A. King.

Non-rusting steel alloys. P. Brès (F.P. 609,277, 11.4.25).—Steels containing cobalt, nickel, manganese, or silicon are heated at 850—1350° in a mass of powdered chromium, ferrochromium, or chromium carbide, or in a mixture of chromium and chromic oxide so that the surface layers contain 9—20% Cr. The articles are subsequently hardened in any suitable way. A. R. Powell.

Extraction of gold from sea water. B. Stoces (E.P. 273,346, 23.12.25).—Sea water is treated with metal sulphides, *e.g.*, antimony sulphide, which combine with or retain gold. C. A. King.

Treatment of bituminous sulphide ores containing copper and zinc. I. G. Farbenind. A.-G., Assees. of C. Müller and W. Schubardt (G.P. 439,171, 31.1.25).—The finely-ground ore is roasted with a limited amount of air so as to drive off the bituminous material and convert the sulphides of zinc and copper into sulphates, which are then leached out with ammonia or ammonium chloride solution. The roasting temperature should not exceed 600° to avoid sintering and the formation of copper matte. The combustible gases evolved from the decomposition of the bituminous matter are used for heating fresh charges of ore. A. R. Powell.

Use of reducing gases in containers made of copper or its alloys. I. G. Farbenind. A.-G. (F.P. 615,441, 30.4.26. Conv., 12.6.25).—The walls of copper vessels are protected from the action of hydrogen by means of a film of inert gas, such as carbon dioxide or nitrogen, which does not reduce cupric oxide. A. R. Powell.

Electrolytic process and apparatus [for depositing metals, *e.g.*, copper]. H. C. Harrison (E.P. 252,320, 5.1.26. Conv., 21.5.25).—A porous cathodic chamber of tubular shape extends longitudinally through an anodic chamber, and is completely enclosed except for the inlet and discharge of the electrolyte at high speed and under pressure. The cathode is of ribbon shape and is kept moving through the chamber by means of rotating external reels. A slow circulation of electrolyte under atmospheric pressure is maintained in the anode chamber. C. A. King.

Furnace for treating zinc ores. A. Roitzheim and W. Remy (E.P. 261,344, 22.9.26. Conv., 13.11.25).—A continuous furnace for the reduction of zinc ores consists of a number of vertical chambers built on supports independently of each other and of the furnace casing. Each chamber is constructed in sections jointed horizontally, and having projections on the outer surfaces on which plates or hollow bricks rest, thus forming horizontal heating flues between the adjacent reduction chambers. The spent material descends into iron pockets which may be water-sealed. C. A. King.

Manufacture of [composite] lead–copper particles. H. M. Williams, Assr. to General Motors Research Corp. (U.S.P. 1,635,653, 12.7.27. Appl., 22.7.25).—Metals which do not alloy readily are mixed in a molten condition and the mixture is atomised when at a temperature higher than that at which the metals tend to segregate. C. A. King.

Hydrometallurgical treatment of oxide products of lead and zinc, or oxidised lead ores containing zinc, or mixed oxidised or sulphide ores of lead and zinc, to obtain therefrom separate metals or concentrated products of lead or of zinc. W. H. Corbould (E.P. 273,420, 13.4.26).—Materials containing lead and zinc oxides are treated with acetic acid to obtain a solution of the metals as acetates, from which lead sulphite is precipitated by the addition of sulphur dioxide. The clear solution may then be freed from copper by replacement with zinc, and zinc may be recovered by electrolysing the neutral solution, treating with hydrogen sulphide, calcium or barium hydroxide, or other appropriate method, acetic acid in every case being regenerated for further extraction. C. A. King.

Extraction of lead and zinc from ores. H. Faivre (Addn. No. 30,609, 10.6.25, to F.P. 596,147; cf. B., 1926, 330).—The ammonia and carbon dioxide evolved in the process described in the chief patent are passed into cold water and the ammonium carbonate solution so obtained is used to convert the lead chloride obtained in the leaching process into lead carbonate with regeneration of ammonium chloride for use in leaching further quantities of lead carbonate ores. A. R. Powell.

Extraction of aluminium ore. A. Finkelstein (E.P. 248,746, 2.3.26).—Aluminium ores are digested with a solution of sodium aluminate of density not higher than 1·36 and the mixture is filtered. The liquor may be diluted with a strong wash liquor from a previous charge to precipitate alumina, and the mother-liquor used either with or without concentration for the extraction of a further quantity of ore (cf. E.P. 13,088 of 1910; B., 1911, 361). C. A. King.

Coating aluminium and its alloys. A. Pacz (E.P. 273,956, 27.11.26).—Aluminium is coated by immersion in a solution containing a silicofluoride, an alkali salt, and a salt of a metal, other than iron, of the iron group. A suitable bath used at temperatures up to 100° contains 0·15% of sodium silicofluoride, 0·2—0·3% of nickel (or cobalt) sulphate, nitrate, or chloride, and 1% of alkali salt. C. A. King.

Making tools from hard metal alloys produced by sintering. F. Krupp A.-G. (E.P. 262,723, 5.8.26. Conv., 12.12.25).—Solid materials produced by compressing a mixture of hard powders, *e.g.*, tungsten or

molybdenum carbides, and softer powders such as cobalt, nickel, or iron, are sintered at 700—1100°, by which treatment they are capable of being filed or ground to the desired shape. They are then hardened by sintering at a higher temperature, *e.g.*, 1300—1600°.
C. A. King.

Casting tungsten carbide and other alloys having high m.p. A. Brennecke (E.P. 259,560, 29.9.26. Conv., 7.10.25).—After fusion in a crucible, alloys of high m.p. are cast directly in a mould connected with the crucible and situated in a part of the furnace having a rather lower temperature than the fusion space. The relative difference in temperature is determined by the properties of the particular alloy. C. A. King.

Working up complex ores and metallurgical products. F. Krupp Grusonwerk A.-G. (E.P. 265,162, 3.1.27. Conv., 26.1.26).—The separation of metals in ores is effected by sublimation of the metal sulphides in a directly heated tube furnace, a neutral atmosphere being maintained during the distillation. Any necessary sulphur can be added if insufficient in the charge, and oxygen may be admitted higher up the furnace to oxidise the sulphide vapours if desired. The sublimation process is controlled so that the sulphides of mercury (400°), cadmium (1300—1400°), arsenic (600—700°), antimony (800—900°), tin and lead (1100—1200°) are distilled off and collected separately from zinc sulphide, copper and ferrous sulphide, etc. C. A. King.

Roasting fine sulphide ores etc. A. L. Mond. From Metallbank u. Metallurgische Ges. A.-G. (E.P. 273,976, 21.1.27).—Fine sulphide ore, *e.g.*, sludge from flotation processes, is mixed with a granular agglomerate of similar origin, which acts as a carrier, and treated by a blast roasting method. C. A. King.

Imparting a close texture to metals. N. N. Jarotzky (E.P. 273,788, 1.4.26).—Molten metal is subjected to centrifugal action in a container capable of rotation firstly on a horizontal axis, and then on a vertical axis. C. A. King.

Metallurgical furnace. J. D. Paton and A. Wood (E.P. 273,457, 3.6. and 3.9.26).—In a furnace using solid fuel, with or without secondary gaseous fuel, one crucible rests on the solid fuel, while a second crucible is supported by a frame above, which is adapted so as to swing the crucible in and out of the furnace. The upper crucible serves as a closure for the top of the furnace.
C. A. King.

Metallurgical furnace plant. B. Talbot (E.P. 273,779, [illegible].3.26).—In a metallurgical furnace having melting baths adjacent to the main hearth (E.P. 235,346; B., 1925, 638), gas and air under pressure are introduced into the auxiliary chambers for the purpose of increasing the temperature, refining the metal, and also raising the temperature of the air supply from the regenerators to the main hearth. C. A. King.

Electric bright annealing furnace. Siemens-Schuckertwerke G.m.b.H. (E.P. 262,463, 3.12.26. Conv., 5.12.25).—In an electric bright annealing furnace into which hydrogen is introduced as protective gas, steam formed during the annealing process is condensed in a cooling chamber provided with water-cooling arranged beneath and communicating with the annealing space. J. S. G. Thomas.

Electroplating with chromium. Chromium Products Corp., Assees. of J. M. Hosdowich (E.P. 258,219, 16.4.26. Conv., 10.9.25).—See U.S.P. 1,590,170; B., 1926, 711.

Method of working shaft furnaces. P. Heskamp (E.P. 272,751, 26.10.26. Conv., 8.9.26. Addn. to E.P. 269,071).

Apparatus for roasting ores (E.P. 254,697).—See II.

Vessels for use with hydrogen peroxide (G.P. 439,834).—See VII.

XI.—ELECTROTECHNICS.

[Crystalline structure of spongy lead, lead peroxide, and lead sulphate] formed during the working of a lead accumulator. L. Mazza (Atti R. Accad. Lincei, 1927, [vi], 5, 688—693).—An *X*-ray examination of the crystal structure of the spongy lead, lead peroxide, and lead sulphate formed during operation on the plates of a lead accumulator has shown that these substances possess the same crystalline structure as their ordinary forms. Spongy lead has lattice constant 4·92 Å. and *d* 11·48. E. A. Lunt.

"Anode effect" in the aluminium bath. K. Arndt (Z. Elektrochem., 1927, **33**, 236—237).—In support of Schischkin's theory of the anode effect (A., 1927, 422) it is noted that in technical aluminium baths, the anode effect occurs more readily when the anode has become coated with finely divided carbon. Also when a normal carbon rod is dipped into an aluminium bath which is allowed to cool until it begins to freeze around the rod, it can be seen that the melt had been drawn up around the rod, *i.e.*, notable "wetting" occurs. In a pure cryolite melt this effect is not observed. If, however, the surface of the rod is previously roughened by heating in the air, it is much less wetted by an aluminium bath and in pure cryolite the melt is seen to be depressed around the rod, like mercury around a glass rod. H. J. T. Ellingham.

Electro-dialysis of sulphite lye. Samec and Ribarić.—See V.

Sterilisation of drinking water. Chevrier and Salles.—See XXIII.

Patents.

Reducing the attack of graphite and carbon electrodes used in the electrolysis of chlorides. Königsberger Zellstoff-Fabrik u. Chem. Werke Koholyt A.-G., and E. Schlumberger (G.P. 439,399, 6.2.25. Addn. to G.P. 437,532; B., 1927, 450).—In the electrolysis of chlorides in accordance with the prior patent, a stream of dilute hydrochloric acid flows through the pores of the electrodes while electrolyte is supplied in the ordinary manner to the electrolyte bath.
J. S. G. Thomas.

Galvanic cell. A. Heil (E.P. 250,266, 1.4.26. Conv., 1.4.25).—In a galvanic cell comprising a zinc electrode and an electrolyte composed of manganese or sodium chloride, the depolariser consists of precipitated or sublimed mercurous chloride rubbed down and

intimately mixed with graphite, pressed under 500—1000 atm. An electrode containing preferably silver or silver alloy, *e.g.*, manganese-silver containing 90% Ag and 10% Mn, or silvered carbon is used. J. S. G. THOMAS.

Double-fluid cell. L. DARIMONT (E.P. 263,081, 20.5.26. Addn. to E.P. 250,803; B., 1926, 551).—In a double-fluid cell of the type described in the prior patent, an alkali salt, *e.g.*, sodium chloride, which diffuses through the porous membrane into the zone of the cement layer between both membranes to make this zone conducting, is added to the depolarising solution of ferrous sulphate or ferric chloride. An organic acid, *e.g.*, tartaric acid, is also added to the depolariser to form an insoluble deposit of calcium tartrate in the cement coating. J. S. G. THOMAS.

Treatment of wooden casings for accumulators. F. KÖNIG (G.P. 438,836, 18.4.26).—The wood is prevented from drying by steeping it, before building up the accumulators, in an organic compound of high b.p. capable of subsequent decomposition by electrolysis into harmless products, suitable compounds including glycol and its lower aliphatic carboxylic or sulphonic acid esters. L. A. COLES.

Electric induction furnace. ELECTRIC FURNACE CO., LTD. From E. F. NORTHRUP (E.P. 274,007—8, 26.4.27).—(A) A cooling medium passes in contact with radiating surfaces in the form of spaced projections or fins provided on the inductor coil of the furnace. (B) An electric induction furnace is provided with a pressure-tight enclosing wall formed principally by a cooled induction coil with sealing and insulating material between its turns. J. S. G. THOMAS.

Electrical purification of gases. SIEMENS-SCHUCKERTWERKE G.M.B.H. (G.P. 439,693, 21.6.25).—The temperature of the gas stream is thermostatically controlled by a bi-metallic strip. The device is applied to maintain the temperature of the tar vapours evolved in the low-temperature distillation of coal above the temperature of condensation of the heaviest constituent of the vapours. J. S. G. THOMAS.

Apparatus for the uniform distribution of the gas stream in electrical gas-purification plants. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of F. MÜLLER (G.P. 439,777, 19.8.25).—Turbulence is produced in the gas stream by means of flat or roof-shaped distributors arranged between the electrodes. For this purpose, the dust-collecting pockets and the discharge and precipitating electrodes are arranged to function as distributors, means being provided for altering the slit width between the distributors. J. S. G. THOMAS.

Desulphonation of anthraquinonesulphonic acids (E.P. 273,043).—See IV.

Frosting glass (E.P. 273,046).—See VIII.

Electrolytic process and apparatus (E.P. 252,320). —See X.

XII.—FATS; OILS; WAXES.

Ultra-violet absorption spectra of some fatty oils. E. LEWKOWITSCH (J.S.C.I., 1927, **46**, 195—197 T).—The ultra-violet absorption spectra of olive, tea-seed, whale, and herring oils showed fairly steep, smooth absorption curves approximating ultimately to the vertical; whale oil showed traces of an absorption band at λ3350 Å., and limiting absorption at about 3500 Å. Four olive oils, varying in colour from yellow to water-white, showed a band or inflexion of the curve at 2750 Å. Admixture of tea-seed oil increased the opacity of the oil to ultra-violet light, but did not suppress the band. The spectrum of chlorophyll in alcohol solution (cold-extracted from dried nettles) showed two distinct absorption maxima at 4210 and 3300 Å., but none to account for the absorption of the olive oils at 2750 Å.

Changes in olive oil after long keeping. R. O. BROOKS (Oil and Fat Ind., 1927, **4**, 139—140, 150).—The history of an old sample of olive oil should be ascertained before condemnation as adulterated. Values recorded in 1906, 1916, and 1926, respectively, for a sample of California oil are: odour and taste, not rancid, very rancid, extremely rancid; Kreis test, —, positive, very strong; $d^{15 \cdot 5}$ 0·9132, 0·9254, 0·9383; iodine value, 83·6, 75·26, 68·58; saponif. value, 190·8, 199·6, 213·2; acid value, 0·90, 80·4, 15·68; ester value, 189·84, 191·56, 197·52; free fatty acids (oleic), 0·48, 4·02, 7·84%. CHEMICAL ABSTRACTS.

Composition of cottonseed oil from the Upland type of seed. G. S. JAMIESON and W. F. BAUGHMAN (Oil and Fat Ind., 1927, **4**, 131—133).—Values for Upland and Sea Island types, respectively, are: olein 30·5, 35·2; linolin 44·8, 41·7; myristin 0·5, 0·3; palmitin 21·9, 20·0; stearin 1·9, 2·0; arachidin 0·1, 0·6; unsaponifiable matter 0·9, —. CHEMICAL ABSTRACTS.

Production of fat by a species of penicillium grown in sucrose solution. H. H. BARBER (J.S.C.I., 1927, **46**, 200 T).—The penicillium, isolated from moulds growing on starch paste in a textile works, produced a deep red colour when grown on starch or sucrose. The surface growth on a liquid sucrose medium containing inorganic salts yielded on drying and extraction with ether 14% of fat, m.p. 13°, saponif. value 197—204, Hehner value 92·9—91·8; fatty acids, m.p. 36°, solidif. pt. 32°, mean mol. wt. 287—289. The fatty acids were separated into approximately equal proportions of saturated acids of m.p. 50·5°, solidif. pt. 49·0°, and liquid unsaturated acids. The residue then extracted with alcohol yielded a red substance which gave an insoluble lead salt.

Relation between the drying of soap flakes and their content of unsaturated fatty acids. G. AISENBERG (Masloboino Scirovoie Delo, 1926, No. 12).—The percentage of water remaining in soap flakes after treatment for 45 min. in a desiccator depends on the proportion of unsaturated fatty acids. To obtain soap flakes containing not more than 10—12% of water after drying for 45 min., the iodine value must not exceed 51·5—52·5. CHEMICAL ABSTRACTS.

PATENTS.

Splitting fats and oils. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM MEISTER, LUCIUS, & BRÜNING (E.P. 261,707, 7.8.26. Conv., 20.11.25).—A fat or oil

is heated at 100° with water and an aromatic mono- or poly-cyclic sulphuric acid or salt thereof containing in the nucleus besides a methyl or ethyl group, an alkyl, aryl, aralkyl, or alicyclic radical of higher mol. wt. The activity of these sulphonic acids is increased if more highly sulphonated products be removed by stirring with sulphuric acid of moderate (about 70%) strength. The use of the product obtained by treating a mixture of naphthalene and ethyl alcohol with chlorosulphonic acid and fuming sulphuric acid is cited as an example.

S. S. WOOLF.

Maintaining the effectiveness of washing and bleaching powders containing soap and oxygen-evolving compounds. T. BENCKISER, A. REIMANN, and A. REIMANN, JUN. (J. A. BENCKISER), and F. DRAISBACH (E.P. 273,414, 7.4.26).—The addition of an alkali hydrogen pyrophosphate, a neutral alkali pyrophosphate, or ammonium phosphate to soap powders containing a perborate or other per-salt, with or without the addition of soda, counteracts the dissociation of the per-salts when the powder is dissolved, without affecting the cleaning action of the soap. S. S. WOOLF.

Manufacture of soap powder. INDUSTRIAL SPRAY-DRYING CORP., Assees. of R. L. HOLLIDAY (E.P. 269,516, 1.4.27. Conv., 19.4.26).—See U.S.P. 1,621,506; B., 1927, 339.

Ferric sulphate (E.P. 273,883).—See VII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Capillary analysis and its application to the testing of resins by Stock's method. E. FONROBERT and K. PISTOR (Farben-Ztg., 1927, **32**, 2424—2429).—The method of differentiating between resins by examination of their "adsorption pictures" (cf. B., 1926, 679) is shown to be limited in scope in its present state of development, the influence of adulterants being insufficiently marked, in general, for the method to attain extended usage. Descriptions of the results obtained with a considerable number of spirit-soluble resins and resin mixtures are given, and criticism and developments of Stock's original experimental details are offered.

S. S. WOOLF.

Physical properties of kauri gum. P. W. BURBIDGE and W. A. MACKY (New Zealand J. Sci. Tech., 1927, **9**, 2—3).—The following data were obtained for kauri gum, using specimens cut from a block of translucent, light amber-coloured gum of d 1·0445 :—resistivity $1 \cdot 41 \times 10^{17}$ ohm-cm., surface resistance $1 \cdot 70 \times 10^{14}$ ohms, dielectric constant 4·7, n_D 1·540, and sp. ht. 0·458. S. S. WOOLF.

Resins formed by condensation of phenol with aromatic hydroxy-aldehydes. J. E. DRIVER (J.S.C.I., 1927, **46**, 197—200 T).—Contrary to popular belief, the resin produced during the Reimer–Tiemann synthesis of *o*- and *p*-hydroxybenzaldehydes is not formed by condensation of phenol with the hydroxy-aldehydes in the alkaline liquid. In presence of aqueous mineral acids, phenol condenses with *o*- and *p*-hydroxybenzaldehydes in approximately equimolecular proportions, forming complex, red, alkali-soluble resins of empirical formula $C_{13}H_{10}O_2$. The salicylaldehyde resin forms a colourless reduction product $(C_{13}H_{12}O_2)_n$, and an acetyl derivative, $(C_{13}H_{10}O_3Ac_2)_n$, of high mol. wt. The respective resins are probably formed by polymerisation of the quinonoid anhydrides of 2 : 4′- and 4 : 4′-dihydroxybenzhydrols. When the resins are impregnated with a solution of an alkali halide, and allowed to dry, the salt separates from the surface of the resin in long, needle-like crystals. A similar change in crystal habit has been observed during the crystallisation of salts from silica gel, and may be taken as an indication of molecular complexity.

PATENTS.

Production of titanium-containing pigments. F. G. C. STEPHENS, L. J. ANDERSON, and W. A. CASH (E.P. 273,017, 26.3.26).—The colour, brilliance, and covering power of titanium white pigments consisting essentially of titanic oxide and an alkaline-earth sulphate are improved by the incorporation with the pigment of boric acid or a suitable borate. S. S. WOOLF.

Preparation of varnishes. I. G. FARBENIND. A.-G. Assees. of L. ROSENTHAL (G.P. 439,009, 30.1.24. Addn. to G.P. 383,699; B., 1924, 265).—Cellulose acetate containing more than 50% of acetic acid is dissolved in a mixture of ethylenechlorohydrin or monochlorohydrin with water, the total water content, inclusive of that in the cellulose acetate, not exceeding 50% of the amount of chlorohydrin present. S. S. WOOLF.

Chlorinated-rubber varnish. N. BOEHMER, Assr. to CHADELOID CHEM. Co. (U.S.P. 1,635,812, 12.7.27. Appl., 26.8.24).—A clear, stable solution of chlorinated rubber is incorporated with boiled drying oil, the latter being boiled to the maximum degree compatible with miscibility. S. S. WOOLF.

Preparation of orange shellac in the wet way. N. N. LAN (F.P. 613,517, 17.6.25).—Mechanically comminuted stick-lac is treated with ammonium carbonate solution, washed, bleached with sodium hypochlorite, neutralised with sulphur dioxide, and dried. The dry product is dissolved in alcohol containing a little ether, and treated with sulphur dioxide or arsenic trisulphide. The shellac is recovered by filtration and evaporation of the solvent under reduced pressure.

S. S. WOOLF.

Manufacture of ozonides of hydrocarbons [turpentine substitutes]. A. S. RAMAGE (E.P. 273,832, 13.4.26).—Products suitable for use as substitutes for turpentine and linseed oil are obtained by ozonising below 125°, and preferably 30—40° below b.p., an olefine mixture or fraction of b.p. 125—250°. If pure amylene, b.p. 35°, is used, refrigeration is necessary. A "light" product, *A*, may be obtained by condensing the vapours evolved during ozonisation; a "heavy" product, *B*, results when ozonisation is continued until the density reaches 1·16. When *B* alone is desired the vapours of *A* are returned by a reflux condenser to the reaction vessel. Product *A* gives alone a hard film in about 6 hrs. and dries linseed oil three times as rapidly as turpentine does. Product *B* is a substitute for linseed oil and when mixed with red lead etc. hardens in the air in 5—6 hrs. C. HOLLINS.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Latex and rubber from young trees. R. Riebl (Med. Proefstat. voor Rubber, 1927, [12], 1—14; Arch. Rubbercultuur, 1927, **11**, No. 4).—The quality of rubber from trees about 8 years old, grown at 2000 ft. above sea level, is considerably better than that of rubber from the same trees at an age of 3—4½ years, but inferior to that of rubber from trees of the same age grown at a lower level. D. F. Twiss.

XV.—LEATHER; GLUE.

Volumetric evaluation of enzymic bates used in tanning. J. Schneider and A. K. Vlček (Chem. Listy, 1927, **21**, 266—269, 286—291).—A known volume of enzymic bate is added to a 9% solution of caseinogen in caustic soda p_H 8·4 and the whole kept for one hour at 40°. Undigested caseinogen is then precipitated by acidifying with hydrochloric acid to p_H 4·5 and adding magnesium sulphate. Formaldehyde is then added to bind amino-groups and ammonia, and the acidity determined by titration with $N/10$ caustic soda, using *o*-cresolphthalein as indicator. This acidity is compared with that obtained using a standard enzyme preparation. R. Truszkowski.

Rapid viscosimeter for glue and gelatin and its application in industrial practice. G. Günther (Chem.-Ztg., 1927, **51**, 526—527).—The Klever rapid viscosimeter consists of a water bath in which is placed a large test tube with a metal cover through which are inserted a thermometer and a measuring pipette with two graduations the volume between which is 30 c.c. The lower end of the pipette is provided with a metal capillary and the upper end with a rubber tube and pinchcock. To carry out a determination of the viscosity of a glue or gelatin preparation, a quantity of the liquid is placed in the test tube and heated at the desired temperature. The pipette is then filled by opening the pinchcock and applying suction to the open end of the rubber tube. When the whole apparatus is at the correct temperature the pinchcock is opened again and the time taken for 30 c.c. of the liquid to flow through the capillary is measured with a stop-watch. The viscosity is then read from a table supplied with the apparatus. The results agree closely with those obtained with the Engler apparatus, but a determination occupies only one tenth of the time. A. R. Powell.

XVI.—AGRICULTURE.

Factors affecting evaporation of water from soil. II. Discontinuity of the drying process. E. A. Fisher (J. Agric. Sci., 1927, **17**, 407—419; cf. B., 1923, 617A; Keen, Crowther, and Coutts, B., 1926, 292).—Further investigations of the drying process of a number of different materials have led to the correction of certain deductions drawn from rate curves previously obtained. The character of the drying system itself is of much greater importance than the environmental conditions in determining the type of curve expressing the rate of loss of water. The drying of any capillary system can be expressed by linear and discontinuous rate curves provided that the movement of moisture or of vapour through the drying mass is regular and uniform; but, if such movement is slow and irregular, as with wet clays and soils, rate curves of various shapes may result and replication becomes impossible. Of the environmental conditions, the most important factors affecting the rate of loss of water are the diffusion of water vapour through the air, and bulk air movements due to temperature gradients between different parts of the drying vessel and to the lowering of temperature of the drying mass itself by evaporation. C. T. Gimingham.

Rapid approximate method for determining soil organic matter. C. J. Schollenberger (Soil Sci., 1927, **24**, 65—68).—The sample (0·5 g.) of air-dried and finely crushed soil is placed in a test-tube with 10 c.c. of chromic acid solution, and heated, with stirring, over a low flame until a temperature of 175° is reached (about 90 sec.). The tube is cooled in water and the contents are poured into 150 c.c. of water, the washings of the tube being added. A few drops of diphenylamine solution are added and the liquid is titrated with standard iron solution. The chromic acid solution consists of 20 g. of potassium dichromate dissolved in 1 litre of concentrated sulphuric acid. The clear liquid is kept for a week and decanted. 0·2N-Ferrous ammonium sulphate is used for titration and the indicator is prepared by dissolving 0·5 g. of diphenylamine in 100 c.c. of sulphuric acid and pouring the mixture into 20 c.c. of cold water. Blank titrations should be made. Results are sufficiently accurate to be used as an approximate measure of the nitrogen content of soils of similar type and known carbon : nitrogen ratio. A. G. Pollard.

Seedling plant method of determining soil nutrient deficiency. J. W. Ames and R. W. Gerdel (Soil Sci., 1927, **23**, 455—466).—Limitations of the applicability of Neubauer's seedling method are examined. The availability of nutrients in the soil varies with the plant type and no one plant can be used for general purposes. Variations occurring between duplicate experiments are frequently so wide as to jeopardise the accuracy of the results obtained. Differences in potash absorption shown by the seedling method are not great enough to serve as a measure of the potash requirement of soils. Contrary to Neubauer's results, no variation in phosphate absorption was observed. The physiological limitations of the method are considered too great to permit its use for the general determination of the availability of mineral nutrients in soils. A. G. Pollard.

Carbon dioxide evolution of soil and crop growth. H. Lundegårdh (Soil Sci., 1927, **23**, 417—454).—Apparatus designed for the sampling and analysis of the soil air is described. A general parallelism exists between the rate of soil respiration and the crop yield on any one soil, but with soils of different type no strict comparison is possible. Fertilisation increases soil respiration, and thus produces an indirect fertiliser action by raising the carbon dioxide content of the atmosphere immediately in contact with the plant leaves. This carbon dioxide fertilisation may be limited by the increased carbon assimilation of the directly fertilised plant, its effective value varying with the nature of the crop. A. G. Pollard.

Influence of calcium cyanamide on the reaction of the soil. J. Pien (Compt. rend., 1927, **185**, 220—222).—Eight samples of soils were mixed with calcium cyanamide and moistened. In all cases addition of the cyanamide caused at first an increase in alkalinity, reaching a maximum in 5—10 days; thereafter, a slight decrease in alkalinity took place until at the end of a month the soils attained equilibium. The soils rich in humus and colloids underwent the more marked variations. B. W. Anderson.

Effect of hydrogen-ion concentration on the growth of certain plants. W. L. Powers (Soil Sci., 1927, **24**, 1—8).—A number of plants examined were found to produce maximum growth in slightly acid media, the optimum range being between p_H 5 and 6 in most cases. For lucerne the optimum range of reaction for nodule production was wider than that for maximum plant growth. Fertiliser salts tending to produce an acid reaction increased the crop yield. Optimum growth range in culture solutions corresponded with that in soils. A. G. Pollard.

Utilisation of phosphoric acid in superphosphate by maize. H. J. Harper, W. G. Baker, B. Boatman, and J. L. Boatman (Soil Sci., 1927, **24**, 9—16).—Fertiliser trials with maize grown on the ridge showed that in soils poor in phosphorus, more than 75% of the phosphate of added superphosphate may be taken up by one crop. Maximum absorption of phosphate occurred in the zone of greatest root development, which was within 6 in. of the centre of the ridge. The intake of phosphate by the plants was not increased by the use of ammonium sulphate and potassium chloride with the superphosphate. A. G. Pollard.

Assimilation of phosphorus from phytin by red clover. A. F. Heck and A. L. Whiting (Soil Sci., 1927, **24**, 17—29).—The phosphorus of phytin is easily and quickly utilised by red clover. The presence of this and similar compounds in the residues of oat crops represents a valuable source of phosphorus for the succeeding crop. Immediate benefits resulting from the use of rock phosphates are attributed to the organic phosphorus therein contained. Lime causes a transformation of easily available organic phosphorus into a less available inorganic form. A. G. Pollard.

Patents.

Preparation of manure. C. F. Boehringer & Soehne, G.m.b.H. (E.P. 268,744, 10.3.27. Conv., 3.4.26).—Cacao bean shells, after treatment with water and slaked lime containing magnesia, are separated from the lye containing the theobromine and part of the tannin and allowed to ferment, with addition of suitable bacteria cultures if necessary, for several months. F. R. Ennos.

Non-caking fertiliser. I. G. Farbenind. A.-G. (F.P. 609,723, 21.1.26. Conv., 9.2.25).—Ammonium nitrate and other deliquescent nitrates, alone or in admixture with other fertiliers, such as ammonium sulphate, are prevented from caking by admixture with calcium monohydrogen phosphate. L. A. Coles.

Fertiliser. Zuckerfabr. & Raffinerie Aarberg A.-G., and A. Rölz (Swiss P. 116,999, 5.6.25).—A mixture of molasses vinasse with superphosphate and material capable of absorbing water, such as peat dust, is simultaneously ground and dried, the proportions being such that the product contains 7—10% of water-soluble phosphoric acids, 2—3% N, 4—7% K, and 30—50% of organic material. L. A. Coles.

Water-soluble phosphatic fertiliser. Préparation Industrielle des Combustibles, and A. Hoffmann (F.P. 614,944, 8.4.26).—Proportionately large quantities of potassium chloride, sodium chloride, or sylvine are added to a filtered solution of crude phosphate in hydrochloric acid, and the solution, after partial evaporation, is cooled to precipitate calcium chloride. The residual liquor is concentrated so that phosphates or a mixture of these with alkali chlorides crystallise out, or is evaporated to dryness to obtain material rich in alkali chlorides. L. A. Coles.

Means for combating plant pests. Chem. Fabr. auf Actien (vorm. E. Schering), Assees. of K. Görnitz and H. Goebel (G.P. 439,077 and 439,365, 26.10.24).—(a) Wash oil residues obtained in the recovery of benzene or (b) liquid coumarone resins are used, alone or in admixture with other fungicides and insecticides, in the form of an emulsion or worked up to a powder with innocuous material such as kieselguhr and talc. L. A. Coles.

Means for combating plant diseases. Chem. Fabr. auf Actien (vorm. E. Schering), Assees. of K. Görnitz and H. Goebel (G.P. 439,235, 18.10.24).—Copper–xanthic acid compounds in concentration not exceeding 1% are used, alone or in conjunction with moistening agents and adhesives and other insecticides and fungicides, such as nicotine. L. A. Coles.

XVII.—SUGARS; STARCHES; GUMS.

Determination of sugar in the beet by the [alcohol] extraction method. A. Dolinek (Z. Zuckerind. Czechoslov., 1927, **51**, 499—511).—Although the alcohol extraction method is regarded as being the only correct one (and is adopted as the standard in comparative tests), it is subject to several sources of error. Under normal conditions, results within 0·2—0·3% of the truth are impossible to obtain, due mainly to the long-continued heating and to the effect of the basic lead acetate, both of which change the rotation of the sucrose and of certain non-sugars. The alcohol extraction process therefore is not recommended, the cold aqueous digestion process carefully applied being far more accurate, besides being quicker and cheaper. J. P. Ogilvie.

Improvement of the Steffen process of extracting sugar from beet molasses. J. Bergé (Sucr. Belge, 1926, **46**, 1—10).—By effecting the addition of lime to the diluted molasses on a layer of liquid free from foam, and by agitating in a device having arms revolving at the rate of 500 r.p.m., a cake of saccharate can now be obtained free from particles of lime, so that instead of a lime consumption of 125—150% of the sugar in process, one of 70—80% is now realised. In washing the saccharate cake, the formation of cracks and channels is rectified by using a dilute suspension of saccharate, instead of water; a saccharate is thus obtainable which on carbonating will give a juice of 17° Brix and 98° purity. J. P. Ogilvie.

Adsorption effect of carbons. J. TRAUBE (Z. Ver. deut. Zuckerind., 1927, 355—364).—Adsorption determinations, using the stalagmometer, show results for commercial carbons in the following order: (*a*) in respect of *iso*amyl alcohol (0·125*N*-solution): Supra-Norit 5, Supra-Norit 4, Merck's *carbo medizinalis*, chlorocarbon, Supra-Norit 2, Kahlbaum's blood carbon, and Carboraffin; (*b*) in respect of sugar (5% solution), diethylamine, *iso*valeric acid, tartaric acid, and other non-colloidal substances: Supra-Norit 5, Kahlbaum's blood carbon, Carboraffin, and bone black; and (*c*) in respect of colloids, *e.g.*, peptone and casein solutions (1%): Supra-Norit 5, Carboraffin. Using beet factory juices, syrups, solutions of raw sugars, and molasses, etc. the results were in the same order as for *iso*amyl alcohol, excepting for *carbo medizinalis*, Supra-Norit 5 being shown to be 1½—2 times as active as Carboraffin. When large amounts of the carbons are used (*e.g.*, above 5%), the differences between them progressively diminish. Supra-Norit 5 and Carboraffin each appear to exhibit different selective effects on colouring substances, being differently charged, and better results are obtained with a mixture of equal parts of the two than with the same weight of either separately. J. P. OGILVIE.

Adsorption of activated carbons. O. SPENGLER and E. LANDT (Z. Ver. deut. Zuckerind., 1927, 429—473).—Using a König–Martens polarisation spectro-photometer for the measurement of the colour, and "Standard Norit" as the standard, the comparative value of some of the carbons examined was as follows: Superior-Norit, 1·1; Eponit-special, 1·6; Supra-Norit 2, 1·6; Carboraffin, 2·7; Supra-Norit 3, 3·2; and Supra-Norit 5, 4·6. Comparisons of the results with molasses and affined sugar solutions showed the utilisation of the carbons to be inferior in the latter case, the presence of the high amount of sugar hindering decolorisation (cf. Bradley, J.S.C.I., 1919, 396 T). Surface tension determinations failed to produce any useful result.
J. P. OGILVIE.

Composition of lime used in the sugar industry. I. A. KUKHARENKO (Nauch. Zapiski [Russia], 1927, **4**, 150—158).—An excess of silica is injurious in the defecation process, and impedes slaking. The presence of magnesia causes the production of a dense, pasty sediment in defecation and saturation. Gypsum is undesirable on account of its solubility in the juices.
CHEMICAL ABSTRACTS.

Crystallisation of sugar from strongly supersaturated solutions. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1927, **51**, 401—412).—An apparatus for determining the viscosity of highly supersaturated solutions is described, by means of which it is shown that crystallisation in such solutions of sucrose begins only when the temperature of the solution exceeds a certain limit, it being therefore possible to have a pure concentrated solution of sugar which fails to crystallise at a low temperature. A definite range of temperature exists within which crystallisation is capable of taking place, this being controlled on the one hand by the temperature at which the solution ceases to be supersaturated, and on the other by the temperature corresponding to the viscosity at which crystallisation may begin. This range is controlled by the composition of the solution, especially its content in positive and negative molasses-forming constituents, which affect the solubility of the sucrose and therefore its saturation.
J. P. OGILVIE.

Identification of sucrose in the presence of invert sugar and other carbohydrates. J. SCHLEMMER (Z. Zuckerind. Czechoslov., 1927, **51**, 422—427).—In identifying traces of sugars in solutions, the liquid under examination is heated in a boiling water-bath for 10—12 min. with 10% of calcium hydroxide, after which the α-naphthol ring test is applied, using 3—5 drops of the reagent in 10—15% alcoholic solution, only 5—8 drops of the liquid, and 2 c.c. of pure concentrated sulphuric acid, added by means of a pipette. Sucrose and raffinose and reversion products then give a positive reaction, whereas dextrose, lævulose, lactose, maltose, starch, dextrin, insulin, and glycogen act negatively.
J. P. OGILVIE.

Tests on the relative sweetness of sucrose and lævulose. J. J. WILLAMAN (Z. Ver. deut. Zuckerind., 1927, 365—367).—Spengler and Traegel's method of establishing the sweetening power of lævulose as 108 and sucrose as 100 (cf. B., 1927, 311) involved remembering how sweet a solution is until the next solution is tasted; the author's procedure is to determine the lowest concentration detectably sweet to a number of experimental subjects while gradually diluting the solutions. This gives a much higher value for lævulose than was stated by Spengler and Traegel, viz., 173, with sucrose as 100. J. P. OGILVIE.

Formation and nature of molasses. J. DĚDEK (Chem. Listy, 1927, **21**, 253—261, 291—297, 317—323; cf. B., 1927, 423).—The velocity of crystallisation of sucrose from its mutually saturated solutions with various salts is least with potassium carbonate, acetate, phosphate, and iodide, sodium trichloroacetate, formate, and acetate, and lithium chloride. Whilst the depression of f.p. observed in various sucrose–salt solutions does not indicate the formation of salts of sucrose, p_H measurements show that the acidic nature of sucrose is augmented more in the presence of salts of inorganic than of organic acids. The viscosity of saturated sucrose–salt solutions, while influencing the velocity of crystallisation, is not a deciding factor in the formation of uncrystallisable solutions. The formation of stable uncrystallisable sucrose–salt solutions is determined by a peculiar shape of the area of solubility obtained in a three-component phase diagram; such conditions exist when the concentrations of sucrose and of sodium and potassium ions are equimolecular. Cane molasses resembles very closely beet molasses, except that the degree of exhaustion of sucrose is smaller. R. TRUSZKOWSKI.

Quality of activated carbons. VYTOPIL.—See II.

Fermentation of cane molasses. KAYSER.—See XVIII.

Fodder tests with sugar etc. JAGODA.—See XIX.

PATENTS.

Diffusion plant for making extracts from sugar beet, sugar cane, etc. C. CAMUSET (E.P. 258,849, 2.9.26. Conv., 25.9.25).—Plant for the continuous

diffusion of cane, beet, etc. comprises a recipient for receiving and heating the product to be extracted, and withdrawing the concentrated liquid, a first vertical, slightly conical diffuser tapering upward in which the product progresses from the top towards the bottom, and a second vertical, slightly conical diffuser tapering downwards, in which the product proceeds from the bottom towards the top, this second diffuser receiving the diffusing liquid and discharging the exhausted product, one or more separate heating devices being provided to maintain the temperature in the different regions of the plant at predetermined values.

J. P. OGILVIE.

Extraction of wax from filter-cake or residue of cane sugar manufacture. T. McKENNA. From H. B. DUNMAN and H. B. LEGG (E.P. 270,036, 24.2.26).—Filter-cake or like residue of cane sugar manufacture containing wax is macerated with sea water, its artificial equivalent, or a solution containing sodium or potassium carbonate, bicarbonate, diborate, or silicate, the mixture being heated, evaporated to dryness, and extracted with a suitable solvent, and the resulting solution saturated with chlorine dioxide. The solvent is subsequently distilled off.

J. P. OGILVIE.

Manufacture of icing sugar. A. E. ACKERS (E.P. 270,822, 16.2.26).—Sugar is pulverised, but, before packing, in order to prevent caking, it is cooled in a suitable manner, *e.g.*, under the action of a circulating current of air.

J. P. OGILVIE.

Manufacture of [prepared] starch. D. R. NANJI (E.P. 273,481, 23.6.26).—Starch is washed with water and, if necessary, digested with pepsin in the presence of hydrochloric acid. A cream of the starch and hydrochloric acid is then treated with potassium permanganate or dichromate etc. After 24 hrs. excess of the oxidising agent is removed by addition of sulphurous acid etc., and the product washed and dried. Solid starch thus treated is mixed with not more than 0·3% by wt. of an alkaline carbonate, hydroxide, borate, etc., the amount used being the minimum necessary to give rise on treatment of the product with water to a clear, substantially neutral solution. The viscosity of the solution varies inversely as the quantity of alkaline agent present. The prepared starch is useful as a sizing agent.

B. FULLMAN.

Fertiliser (Swiss P. 116,999).—See XVI.

XVIII.—FERMENTATION INDUSTRIES.

Vitamin content of beer. A. SCHEUNERT and M. SCHIEBLICH (Biochem. Z., 1927, **186**, 229—231).—A sample of black beer is shown by experiments with rats and pigeons to contain only a very small amount of vitamin-*B*, an amount comparable with that previously found in other kinds of beer and smaller than that in porter.

P. W. CLUTTERBUCK.

Sulphur dioxide in wine making. L. MOREAU and E. VINET (Ann. Falsif., 1927, **20**, 316—325).—Addition of sulphur dioxide to a must or a wine results in complete combination with certain constituents—aldehydes etc.—up to a limiting value, which, expressed in mg. of sulphur dioxide per litre (index *T*), is characteristic of the must or wine at the time of examination. For any subsequent addition the combination is only partial, a definite fraction of the added sulphur dioxide remaining in the free state. Index *R* is the proportion of sulphur dioxide remaining after each such addition of 100 mg./litre. The indices of combination are determined from the straight-line curve expressing the relationship between the sulphur dioxide added and that remaining in the free state after combination. Two methods are described, one of which takes 4 days to complete, the other, which is useful when a rapid approximate figure is required, occupying only 4 hrs. The index *T* of a series of musts and wines examined varied between 50 and 170 for the former and between 30 and 205 for the latter. Index *R* for the musts varied between 42 and 56, but for wines its value was practically constant at 75. From a knowledge of *T* and *R* it is possible to calculate the amount of sulphur dioxide which should be added to a must or a wine to give it any desired content of free sulphur dioxide, which is the active germicidal agent.

F. R. ENNOS.

Colour and alcohol content of dutiable wines. P. STADLER (Z. Unters. Lebensm., 1927, **53**, 340—341).—Attention is drawn to the difficulty of making a sharp distinction between red and white wines, which, according to recent customs regulations, are liable to different rates of duty. White wines containing less than 140 g. of alcohol per litre are subject to higher duty than wines richer in alcohol, and it is shown that determining the specific gravity to four decimal places instead of five as formerly, may place in the higher dutiable class, wines which strictly belong to the lower. The application of a correction to wines having an acid content of over 1·2 g. per litre has a similar influence. Wines of low alcohol content are being adulterated with spirit in order to bring them into the class of lower duty.

H. J. DOWDEN.

Determination of hydrogen-ion concentration of wine. R. DIETZEL and E. ROSENBAUM (Z. Unters. Lebensm., 1927, **53**, 321—330).—The acidity of wine can be conveniently determined by measuring its p_H by means of the quinhydrone electrode. The *E.M.F.* of the system Pt | 0·1 g. quinhydrone, 0·01*N*-HCl | sat. KCl soln. | 0·01*N*-HCl, H_2 | Pt was measured against a standard Weston element by the compensation method, from which a value of 701·8 millivolts was found for the cell. Taking the *E.M.F.* of the calomel electrode against a normal hydrogen electrode as + 250·3 millivolts, the *E.M.F.* of the quinhydrone electrode in a normal hydrogen-ion solution against the calomel electrode is 452·5 millivolts, allowing 1·0 millivolt for the potential of the hydrogen in the electrode at 760 mm. pressure. The potential of a hydrogen electrode in the wine is obtained by deducting 452·5 millivolts from the *E.M.F.* between the quinhydrone and the calomel electrodes. The hydrogen-ion concentration is given by the equation $p_H = -E/58\cdot1$.

H. J. DOWDEN.

Detection of fruit wine in grape wine. K. MÜLLER, E. VOGT, and O. RAESCH (Z. Unters. Lebensm., 1927, **53**, 331—334).—A valuable indication of the presence of fruit wine in grape wine is given by the colour of the fluorescence exhibited in ultra-violet light, as proposed

by Reich, and especially when neutralised with caustic soda and mixed with about 15% by vol. of fresh ammoniacal copper acetate solution, as proposed by Röttgen. Pure untreated grape wines fluoresce with a greyish colour in ultra-violet light, and when treated with copper acetate, the colour is clear blue. The wines must be filtered, as cloudy wines, especially red wines, show a brown fluorescence. Fruit wines appear brown in ultra-violet light and when treated with copper acetate, olive-green. Mixtures of fruit and grape wines show intermediate colours and the addition of 20% of fruit wine can be detected, provided it has not been treated with clarifying agents. H. J. Dowden.

Detection of fruit wine in grape wine. M. Rüdiger and W. Diemair (Z. Unters. Lebensm., 1927, **53**, 335—340).—The reliability of Schaffer and Schuppli's method for the detection of fruit wines by their reducing action has been examined. 50 c.c. of wine were decolorised by shaking with 1 g. of animal charcoal and filtered after ½ hr. After accurate neutralisation of the filtrate with barium hydroxide solution and refiltering, 5 c.c. were mixed with 2 drops of $0 \cdot 2N$-silver nitrate solution and 0·5 c.c. of $0 \cdot 2N$-caustic soda solution. With fruit wines, brownish-black colorations of varying intensities were produced at once, or after a few minutes. With white wines from grapes, the majority remained colourless for ½ hr., the remainder giving pale yellow or red colours. Red wines from grapes needed a larger amount of charcoal for decolorisation and gave inconclusive results. Wines from the marc of grapes all gave more or less strong colorations comparable with those given by fruit wines. Mixtures of grape and fruit wine gave in most cases indication of the adulteration, by more or less dark colorations; the remainder gave no colour. A 0·05% solution of tannic acid gave a coloration identical with that given by fruit wine, but when added to a white wine and treated with charcoal, the reducing properties were removed. The inference is drawn that reducing action is dependent upon the tannin content of the wine and upon the nature of its combination. The method is inapplicable to grape wines rich in tannin.

H. J. Dowden.

Nitrogenous nutrients of yeast in the fermentation of maize and potato mashes. J. Dehnicke and W. Kilp (Z. Spiritusind., 1927, **50**, 202—204).—The amounts of total nitrogen and of amino-nitrogen in a mash prepared from potatoes and malt exceed those in a corresponding mash made from maize and malt. In the maize mash there is approximately two thirds of the total nitrogen and little more than one half of the amino-nitrogen of the corresponding potato mash. The differences are most marked when the proportion of malt present is small. In the potato mash, the nutrients, of which the amino-acids are the most important, are more favourable to the yeast than in the maize mash, although in the original maize there is a higher nitrogen content than in the potatoes. With both mashes the amount of total nitrogen remains fairly constant during the fermentation, but the greater proportion of the amino-acid nitrogen is rapidly consumed by the yeast during the primary fermentation and during the period of yeast reproduction. During the later periods of fermentation the amino-nitrogen content also remains more or less constant. Of the nitrogenous nutrients which may be added to the maize mashes, preference is given to autolysed yeast or dried yeast hydrolysed by sulphuric acid. Ammonium salts should be avoided. C. Ranken.

Fermentation of cane molasses. E. Kayser (Ann. Falsif., 1927, **20**, 326—328).—The amount of higher alcohols in rum is increased by addition to the fermenting molasses of magnesium phosphate, or of a yeast which has undergone autolysis, an anaerobic yeast producing a larger increase than an anaerobic one. The yield of esters obtained at the same time is also considerably increased by the aerobic yeast, to a much smaller extent by the anaerobic one, and is appreciably diminished by addition of magnesium phosphate.

F. R. Ennos.

Production of fat by penicillium. Barber.—See XII.

Benzoic acid. Noetzel.—See XX.

***iso*Propyl alcohol.** Noetzel.—See XX.

Patents.

Manufacture of yeast. A. P. Harrison, Assr. to Internat. Yeast Co., Ltd. (E.P. 259,572, 5.10.26. Conv., 6.10.25).—The apparatus is of the continuous-addition–continuous-withdrawal type consisting of a series of concentrically disposed fermentation compartments connected by constant-level liquid-seal gas-trap outlets with provision for the control of the temperature of at least one compartment. Each compartment contains baffle-dividing means so situated that aeration can take place on each side of the baffle. The seed yeast is suspended in a dilute nutrient solution which is fed into the bottom of the first compartment, and reserve nutrients in solution are added at such a rate that the density in the first compartment remains as nearly as possible constant. In the compartments beyond, the density is also kept equal and constant but somewhat lower. The volume of the liquid is maintained substantially constant.

C. Ranken.

Manufacture of pressed yeast. R. Kusserow (G.P. 439,498, 29.10.22).—Molasses with added sulphuric acid and sulphites is diluted to 25° Balling by the addition of an aqueous suspension of chalk, the liquid being kept cold. Other yeast nutrients are added and the whole is aerated during the fermentation by yeast.

C. Ranken.

XIX.—FOODS.

Interruption of milking and composition of milk. F. E. Nottbohm (Z. Unters. Lebensm., 1927, **53**, 342—353).—As the exhaustion of the milk supply proceeds, the changes in composition vary with different cows. In some cases but little change occurred, whilst in others the lactose decreased from 5 g. to about 1 g. and the chlorine content increased from about 0·1 g. to 0·2 g. per 100 c.c., whilst at the same time considerable changes in the colour, taste, and acidity of the secretion were observed. In the same period the casein-nitrogen varied fron 82% to 45% of the total nitrogen. It is therefore recommended that milk obtained during the "drying-off" period or after an interruption of the

customary twice-a-day milking should be prohibited from being sold as "fresh" milk. H. J. Dowden.

Determination of the butyric acid value [of milk fat]. J. Grossfeld (Z. Unters. Lebensm., 1927, **53**, 381—383).—To obviate the necessity of weighing and dissolving 15 g. of sodium sulphate for the "salting out" of butyric acid in the method described by Kuhlmann and Grossfeld (cf. B., 1926, 447), a saturated solution of the salt was used, but difficulty was experienced owing to the formation of insoluble sodium soap curds. Potassium soaps being more readily soluble did not give rise to this difficulty and accordingly 150 c.c. of saturated aqueous potassium sulphate solution were used in the place of the 15 g. of anhydrous sodium sulphate. The results thus obtained were in good agreement with those yielded by the sodium sulphate method. H. J. Dowden.

Ewe's milk and its adulteration. A. Sanna and N. Baiardo (Annali Chim. Appl., 1927, **17**, 297—307).—When the f.p. depression of ewe's milk is below 0·59°, the other analytical results being those of the genuine milk, the addition of 10—15% of cow's milk is indicated. With greater proportions of added cow's milk, the judgment is aided by the diminution in the percentages of casein and fat and by the normal values of the density of the milk and of the whey; moreover, the cryoscopic value then approaches that of cow's milk, but other constants are changed only slightly. Adulteration by addition of whey involves lowering of the f.p. and decrease of the dry residue of casein, which becomes lower in proportion to the fat; the densities of the milk and whey, and the amounts of ash, albumin, and lactose remain unchanged. Addition of water is detected by its diluting effect. T. H. Pope.

Cod-liver oil as food. Existence of vitamin-*E*. V. E. Nelson, R. L. Jones, G. Adams, and L. F. Anderegg (Ind. Eng. Chem., 1927, **19**, 840—844).—It is thought that vitamin-*E* is required for normal reproduction in animals and that in its absence they become sterile. Reproduction with rats is possible but not normal on a synthetic diet of casein, salts, filtered butter fat, yeast, and dextrin, and it is considered that butter contains small amounts of vitamin-*E*. The food value, as regards growth and reproduction, of cod-liver oil incorporated in synthetic diets as the sole source of fat-soluble vitamins, was investigated, a diet of casein, salts, cod-liver oil, yeast, and dextrin being administered to rats. The reproduction results were much superior to those using butter fat, but varied with the manner in which the oil was administered, incorporation with large quantities of the diet being much less favourable owing to decomposition of the oil with the other constituents when kept. W. G. Carey.

Wood light for detecting soya bean flour when mixed with wheat flour. G. Cappelli (Annali Chim. Appl., 1927, **17**, 308—312).—Ultra-violet rays from a quartz mercury vapour lamp are incapable of detecting the presence of soya bean flour in wheat flour. When subjected to Wood light, however, raw soya bean flour exhibits a brilliant, greenish-yellow olour and the defatted flour a greenish-yellow or canary colour. Under the same light, Italian 82—84% wheat flour shows a shining, slightly nacreous, scaly appearance with a characteristic bluish-violet fluorescence. This fluorescence is attenuated by the presence of 1—4% of soya bean flour, and is replaced by the canary-yellow coloration almost completely when 10% of soya bean flour, and entirely when more than 10%, is present. T. H. Pope.

Vitamin content of sterilised foodstuffs. E. Remy (Z. Unters. Lebensm., 1927, **53**, 297—306).—Nutrition experiments on rats have been performed to determine whether sterilised and preserved foodstuffs are devoid of vitamins. The materials investigated included green beans, green peas, carrots, spinach, veal, and milk, all of which were sterilised by heating at 98—100° and then stored for 9—10 months. A diet containing a small quantity of vitamin-*B* and consisting of yeast flakes 40·0%, casein 5·0%, sodium chloride 1·0%, calcium carbonate 3·0%, and dextrin 51%, was given to rats for a period of 40—50 days. The lack of vitamins was evidenced by the arrest of growth, diseases of the skin, hair, and eyes, and by rachitic symptoms in the joints. The addition of a small proportion of any one of the sterilised foodstuffs to the vitamin-free diet produced in every case a marked and continuous increase in weight and improvement in condition of the rats. The average daily increase in weight when sterilised peas were added was 6·2% greater than that of the control rat, which was fed on a normal diet. With the other foodstuffs, the daily increase was less than that of the normally fed animal, the difference with carrots and spinach being about 40%. It is therefore concluded that vitamins-*A*, -*B*, and -*D* are present in sterilised foods, even after a comparatively long storage period. H. J. Dowden.

Formation of black mustard essence. Lasausse (Ann. Falsif., 1927, **20**, 328—334).—Mustard flour, when macerated with water at various temperatures, develops the maximum amount of essence after 1 hr., longer periods resulting in a lower yield (cf. Colombier, B., 1926, 606). The amount of essence obtained after any time depends on its rate of formation and destruction, and the influence of various factors on these reactions was therefore examined. Fineness of grain promotes the formation of essence, but addition of myrosine has practically no effect. Excess of mustard essence tends to hinder the rate of essence formation. Maceration with a solution of salt and acetic acid lowers the amount of essence formed after 1 hr., but prolonged treatment gives gradually increasing amounts until a maximum, very nearly equal to that obtained in the absence of acid, is reached. Increasing the acid content of the macerating solution diminishes the essence formed after 1 hr., and increases the time required to reach its maximum development. The addition of acids—acetic or tartaric—to mustard preparations ensures the gradual development of the essence by retarding both its rate of formation and decomposition, and also by hindering the development of destructive bacteria. Loss of mustard essence by evaporation takes place even in oil solutions. F. R. Ennos.

Oven-drying method for the determination of moisture in cottonseed meal. P. Sherrick (J. Oil and Fat Ind., 1927, **4**, 106—111).—Uniform results may be

obtained by using a vacuum chamber in a circulating oil thermostat. The oil bath is controlled to variations less than 0·5° by means of the De Khotinsky bimetallic thermo-regulator, U.S. Bureau of Standards form knife type heaters, and a motor-driven stirrer.

Chemical Abstracts.

Acid- and base-forming elements in foods. G. W. Clark (J. Biol. Chem., 1927, **73**, 389).—In the author's previous paper (B., 1926, 74) the figure for the nitrogen content of fresh eggs should be 1·7%, not 7·767%, which applies to the dried material.

C. R. Harington.

Fodder tests with additions of [beet] raw sugar and molasses, also saccharin. G. Jagoda (Z. Ver. deut. Zuckerind., 1927, 243—331).—In feeding-tests with pigs, additions of beet raw sugar and molasses to rations of fish meal, barley, and potatoes gave, in general, satisfactory results, the animals tolerating 750 g. either of the sugar or molasses per animal per day, and remaining healthy and vigorous during the 11-weeks' test. A gain in weight was obtained sufficient to enable the fattening period to be reduced by 7 days, the nett conclusion being that both forms of carbohydrate are economical for the purpose (in the absence of any tax on sugar). Saccharin (Fahlberg's × 450 times) was tolerated to the amount of 2 g. per animal per day during 4 weeks; but, though the reaction of the animals to this sweetening agent was later and less marked than with sugar and molasses, the ration on the whole was better utilised than in its absence. Its use, however, is considered uneconomical, unless its tax-free sale is permitted.

J. P. Ogilvie.

Cadmium as plating metal for utensils. Gronover and Wohnlich.—See X.

Vitamin content of beer. Scheunert and Schieblich.—See XVIII.

Benzoic acid. Noetzel.—See XX.

Patents.

Treatment of meal, flour, meal- and milling-products. N. V. Internat. Oxygenium Mij. "Novadel" (E.P. 249,139, 11.3.26. Conv., 11.3.25).—The flour etc. is successively injected with solutions, suspensions, or emulsions first of an aldehyde and then of an acid anhydride, after which air or oxygen is blown through. To stimulate the formation of the flour-improving materials a catalyst, *e.g.*, salts of metals of the iron group, and a weakly alkaline material may be added.

F. R. Ennos.

Manufacture of jams, jellies, and other preserves. D. McKinlay (E.P. 273,578, 14.12.26).—The ingredients are subjected to the action of artificially produced ultra-violet rays while being heated in a shallow pan provided with means for automatically agitating the contents and for removing the scum from the surface. F. R. Ennos.

Carbonating milk or cream. D. Sweeney (E.P. 273,835, 14.4.26).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Chemistry in the progress of medicine. F. H. Carr (J.S.C.I., 1927, **46**, 263—270 T).—A review dealing with hormones, antitoxins, organo-metallic compounds, and alkaloids.

Preservation of ether for anæsthesia. F. Bicknese (Pharm. Zentr., 1927, **68**, 439).—Ether for narcosis may be preserved over iron powder, and remains free from peroxide after keeping for two years. Iron powder also removes peroxide originally present. S. I. Levy.

Determination of *iso*propyl alcohol in the presence of ethyl alcohol. O. Noetzel (Z. Unters. Lebensm., 1927, **53**, 388—391).—*iso*Propyl alcohol may be detected by oxidation to acetone by means of chromic acid, the acetone being recognised by the nitroprusside reaction. The presence of *iso*propyl alcohol in ethyl alcohol produces but little change in *d* and b.p., but appreciably raises the refractivity. The determination of *iso*propyl alcohol in wines by the latter method is not practicable owing to the disturbing influence of acids and esters. Oxidation to acetone affords a method of determining *iso*propyl alcohol. The alcohol is diluted so that 100 c.c. contain not more than 5 c.c. of alcohol; 25 c.c. are mixed with 50 c.c. of potassium dichromate solution (96 g./litre), 100 c.c. of 50% sulphuric acid are added, and the liquid is cooled. After 3 hrs. at room temperature, the unchanged chromic acid is reduced by adding 100 c.c. of 25% ferrous sulphate solution and the mixture is steam distilled. 75 c.c. of the distillate are collected in a vessel containing 5 c.c. of 10% caustic soda solution and 15 c.c. of water. The distillate is redistilled, about 50 c.c. being collected in a receiver containing 2 g. of hydroxylamine hydrochloride in 20 c.c. of water, with which the acetone reacts forming acetoxime and liberating hydrochloric acid. After shaking and keeping for 1 hr., the free hydrochloric acid is titrated with 0·25*N*-caustic soda solution against methyl-orange, using a plain hydroxylamine hydrochloride solution as colour standard. The result of the blank experiment is deducted from the titer of the distillate, the weight of *iso*propyl alcohol being then given by the factor 0·015. H. J. Dowden.

Determination of benzoic acid [in foods, wines, etc.]. O. Noetzel (Z. Unters. Lebensm., 1927, **53**, 383—387).—Separation of the benzoic acid is effected by distilling the material with a hydrocarbon such as petroleum, the b.p. of which is higher than the sublimation temperature of the acid. 80 g. of meat are ground with 15 c.c. of 25% phosphoric acid, shaken with 135 c.c. of purified petroleum, and distilled. When the water has been removed, the temperature is raised to 195° and a layer of petroleum of about 105 c.c. is collected. The united distillate is rendered alkaline with lime-water and, after shaking, the aqueous layer is separated, filtered, and purified by the permanganate method of Heide and Jacob (cf. A., 1910, ii, 359). After removal of all manganese by treatment with hydrogen peroxide and filtering, the filtrate is acidified with 2*N*-sulphuric acid and extracted four times with ether, the combined extracts being washed with water, dried, and evaporated to dryness. The crude benzoic acid is weighed and a further determination made by dissolving in an alcohol–ether mixture and titrating with 0·1*N*-caustic soda, using phenolphthalein as indicator. Very small quantities of benzoic acid are best purified

by the Polenske sublimation method. The distillation method is directly applicable to butter, margarine, and egg products, but impracticable with fruit juices etc. owing to the decomposition of the sugars. In such cases the acidified juice is extracted several times with ether, the extracts being shaken with a slight excess of caustic soda and the ether removed by distillation. The residue is then treated with phosphoric acid and distilled with petroleum. Wines not very rich in alcohol or sugar may be distilled with petroleum directly, but sweet wines must be extracted as described for fruit juices. Jams etc. must also be extracted with ether but the results are not quantitative owing to the formation of emulsions. H. J. DOWDEN.

Preparation of atropine and cocaine. F. CHEMNITIUS (J. pr. Chem., 1927, [ii], **116**, 276—290).—A description of methods and apparatus suitable for the extraction of atropine from quantities of 500 kg. of the roots of *Atropa belladonna*. The powdered drug, which should contain at least 0·5% of atropine, is moistened with 10% sodium carbonate solution, dried, and exhausted with ether. Crude atropine is precipitated from a 5% acetic acid solution of the extract by adding potassium carbonate (*a*), recrystallised from ether, and heated to 120° with chloroform to racemise any hyoscyamine present. The mixture is poured into acetone; the crystalline product obtained on cooling (*b*) may be used directly for the preparation of salts, of which the sulphate, hydrochloride, valerate, and salicylate are described. One recrystallisation of the base from acetone gives pure atropine. The mother-liquors, *e.g.*, from (*a*) and (*b*), contain hyoscyamine, which is recrystallised from ether and finally converted into atropine. The determination of cocaine in crude cocaine is described. The product should be white and granular; inferior material is scaly. It is purified by adding permanganate at 0—5° to an acetic acid solution until oxidation is complete. The cocaine is precipitated, together with manganese dioxide, by potassium carbonate and extracted from the dried product with ether, from which it separates in the pure state on concentration. The mother-liquors may be freed from impurities by adding petroleum and the cocaine recovered from the solution as hydrochloride. Non-crystalline residues are hydrolysed to ecgonine, which is reconverted into cocaine. The preparation of cocaine hydrochloride and nitrate is described. H. E. F. NOTTON.

Iodine–bromine values of the ethereal oils. L. W. WINKLER (Pharm. Zentr., 1927, **68**, 433—439).—The iodine–bromine values of the commoner ethereal oils, as determined by the methods of the German and Hungarian pharmacopœias, are tabulated. Generally they show wider variations and are more characteristic than the values for fats and fatty oils, the values ranging from 1—2 (oil of bitter almonds) to 355—365 (oil of orange). The determination is suggested as a simple aid to identification. S. I. LEVY.

Distillation of Java citronella oil in a high vacuum. H. I. WATERMAN, F. J. NELLENSTEYN, and N. P. J. DAAMEN (Rec. trav. chim., 1927, **46**, 509—515).—900 g. of the crude yellow oil having $n_D^{20.9}$ 1·4688 was divided into fractions having b.p. 63°, 63—65°, 65—75°, 75—85°, 85—95° at 2 mm., and 85—90°, 90—100° at 1 mm., and by redistillation of these in a cathode vacuum 292 g. of pure citronellal, b.p. 36°, n_D^{20} 1·4485, and 111 g. of geraniol, b.p. 47—50°, n_D^{20} 1·4756—1·4758, were obtained. On keeping, the citronellal showed an increase in refractive index and sp. gr., although it gave correct figures on elementary analysis. When these constants had increased to $n_D^{19.1}$ 1·4521, d_4^{20} 0·8720, the sample was redistilled in a high vacuum, and citronellal having b.p. 41—47° $n_D^{20.8}$ 1·4481—1·4487, d_4^{20} 0·8580 was obtained, together with a higher fraction, b.p. above 47°, n_D^{20} 1·4598, d_4^{20} 0·9179, which is presumably a decomposition product formed from citronellal on storage. W. J. POWELL.

Black mustard essence. LASAUSSE.—See XIX.

PATENTS.

Catalytic dehydrogenations. I. G. FARBENIND. A.-G. (E.P. 263,877, 3.1.27. Conv., 4.1.26. Addn. to E.P. 262,120; B., 1927, 541).—Instead of using sulphides as catalysts, heavy metal phosphides, selenides, tellurides, antimonides, arsenides, or bismuthides are employed alone or in mixture, such zinc compounds being specially suitable. W. G. CAREY.

Preparation of formic acid derivatives. KOEPP & Co., and E. ELÖD (G.P. 439,289, 10.9.22).—Alkali hydrogen formates are used in place of anhydrous formic acid for the preparation of derivatives. High temperatures may be used although sodium hydrogen formate ordinarily decomposes above its m.p. 66°. The preparation of glyceryl diformate, ethyl formate, phenyl formate, glycol formates, geranyl formate, and mixed anhydrides of formic acid with acetic acid and nitric acid is described. C. HOLLINS.

Manufacture of aldol and crotonaldehyde from acetaldehyde. O. SILBERRAD (E.P. 273,776, 15.11.26).—Acetaldehyde is added slowly to a stirred suspension of barium hydroxide in two thirds its weight of water below 10°. The baryta is then neutralised (with oxalic acid) and the filtered product may be distilled slowly for crotonaldehyde, or in a vacuum for aldol. C. HOLLINS.

Manufacture of symmetrical diarylguanidines. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 262,155, 30.11.26. Conv., 30.11.25).—A salt of an *s*-alkyl- or -aralkyl-thiocarbamide is heated with an arylamine. Diphenylguanidine, m.p. 148—149°, is obtained from aniline and an *s*-ethyl-, *s*-methyl-, or *s*-benzyl-thiocarbamide salt at 100—130°. *Di-o-anisylguanidine*, m.p. 120° (*hydrobromide*, m.p. 195—196°), is similarly prepared. C. HOLLINS.

Production of an iodopyridine. A. BINZ and C. RÄTH, Assees. of DEUTSCHE GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESSLER (E.P. 259,977, 14.10.26. Conv., 17.10.25. Addn. to E.P. 251,578: B., 1926, 608).—3-Aminopyridine is diazotised and then heated with potassium iodide solution to give 3-iodopyridine, m.p. 50°. C. HOLLINS.

Manufacture of acid [*O*-acyl] derivatives of αα′-dimethyl-γ-hydroxypiperidine-β-carboxylic [4-hydroxy-2 : 6-dimethylpiperidine-3-carboxylic] acid esters. I. G. FARBENIND. A.-G. (E.P. 271,467, 16.5.27. Conv., 17.5.26).—*O*-Acyl derivatives which have local anæsthetic properties are obtained by esterification of alkyl 4-hydroxy-2 : 6-dimethylpiperidine-3-carboxylates. Thus the methyl ester, m.p. 143°, is

treated with benzoyl chloride or benzoic anhydride in chloroform to give the *hydrochloride*, m.p. 206°, of *methyl 4-benzoyloxy-2 : 6-dimethylpiperidine-3-carboxylate.*
C. HOLLINS.

Production of heterocyclic arsenic compounds. DEUTSCHE GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESSLER (E.P. 250,577, 1.4.26. Conv., 11.4.25).—Arseno-compounds containing on one or both sides a nitrogen ring residue, prepared by the usual methods, have a higher ratio of tolerated to curative dose than the corresponding arsenobenzenes, the curative dose being in some cases extraordinarily small. 5 : 8-*Arsenoquinoline* is obtained by reduction of a mixture of quinoline-5- and -8-arsinic acids with hypophosphorous acid at 70°; 8-(2-*hydroxy*-5-*pyridinearseno*)*quinoline* and 2-(2-*hydroxy*-5-*pyridinearseno*)*benzoic acid* are similarly prepared. 2-*Hydroxy*-5-*pyridyldichloroarsine*, prepared by treating the arsinic acid with hydrochloric acid, potassium iodide, and sulphur dioxide, is converted by dissolution in water into the *arsenious oxide*, which is condensed with 3-amino-4-hydroxyphenylarsine to give 2-*amino*-4-(2-*hydroxy*-5-*pyridinearseno*)*phenol*, $HO\cdot C_5H_3N\cdot As:As\cdot C_6H_3(OH)\cdot NH_2$, also obtained by reducing a mixture of 2-nitrophenol-4-arsinic acid and 2-hydroxypyridine-5-arsinic acid, or by stirring together a mixture of the two symmetrical arseno-compounds in hot aqueous suspension. The *formaldehydesulphoxylate* derivative of this compound is described. C. HOLLINS.

Manufacture of benziminazolone-5-arsinic acids. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 256,243, 27.7.26. Conv., 31.7.25).—*o*-Phenylenediamine-4-arsinic acids, in which one amino-group carries an alkyl, aralkyl, or alkylenyl substituent, obtained from 3(or 4)-chloro-4-(or 3)-nitrobenzenearsinic acid by condensation with a suitable amine, are treated with carbonyl chloride in presence of aqueous sodium acetate to give products which are more active therapeutically than the unsubstituted benziminazolone-5-arsinic acid. 1-*Methyl*-, 3-*methyl*-, 1-*propyl*-, 1-*allyl*-, and 1-*benzyl-benziminazolone*-5-*arsinic acids* are described, all melting above 280°.
C. HOLLINS.

Manufacture of substances for reducing arterial hypertension. W. J. MACDONALD (E.P. 250,230, 29.3.26. Conv., 3.4.25).—Fresh, fat-free calf liver (or related gland) is chopped with chloretone, ground with sand, and extracted with Locke's solution containing 0·01% of sulphuric acid. The solution is evaporated, the residue extracted with 80% alcohol, the clear solution again evaporated, and the residue therefrom dissolved in physiological salt solution. The solution is finally passed through a Berkefeld filter, treated with a quantitatively equal volume of alcohol, and acetone added. A reddish-brown oil, *B*, separates. On adding more acetone a white solid, *A*, is precipitated. *A* and *B* may be further purified by dissolution in water and precipitation by addition of alcohol and acetone.
B. FULLMAN.

Treating bacteria, their products and toxins, and serums obtained from the treated bacteria or toxins. F. B. DEHN. From E. LILLY & CO. (E.P. 273,349, 4.1.26).—Bacteria or their toxins may be detoxified without loss of their antigenic properties by treating them *in vitro* with surface-tension-depressing substances. The substances used are soluble salts (substantially free from digestive ferments) of fatty acids having m.p. below 44°, preferably unsaturated hydroxylic fatty acids, and especially sodium ricinoleate and castor oil soap. Sodium proteinates are also applicable. The production of anti-diphtheria, anti-scarlet fever, and anti-pneumonia sera is claimed. B. FULLMAN.

Ozonides (E.P. 273,832).—See XIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Influence of pre-exposure and exceptions to the law of reciprocity in photography. J. HRDLIČKA (Compt. rend., 1927, **185**, 120—122).—Assuming the photochemical effect of two successive exposures (t_0 and t sec.) to be the same as that of an exposure of $T=(t_0+t)$ sec., the curves of blackening of an emulsion having undergone a preliminary exposure of t_0 sec. may be constructed from the expression $D=\phi(I\times T)=\psi(I\times t)$, where D is the density of the image, I the intensity of the light, and ϕ and ψ the functions of blackening of the emulsion before and after the preliminary exposure, respectively. Good agreement between theoretical and practical results was obtained when the preliminary and successive exposures were of the same intensity. The increase in density with time is given by the expression $Kt/(t_0+t)$, where K is a constant. J. GRANT.

XXIII.—SANITATION; WATER PURIFICATION.

Determination of hardness and alkalinity of water. H. VIGNAL (Ann. Chim. anal., 1927, [ii], 9, 162—168).—A routine method is described, in accordance with which a sample is first titrated with standard acid, then with soap solution, with the latter again after precipitation of calcium as oxalate, and after precipitation of sulphates by addition of barium chloride, the hardness due to the latter being allowed for, and finally with silver nitrate. By using burettes suitably divided and marked, and standard solutions of suitable concentrations, the various factors may be read off in terms of hardness with the minimum of calculation.
S. I. LEVY.

Sterilisation of drinking water by electrolysis. D. CHEVRIER and M. SALLES (Compt. rend., 1927, **185**, 230—231).—Electrolysis of drinking water, using a chrome-steel or silver cylindrical cathode enclosing an anode of platinum wire and a potential of 110—120 volts, causes efficient sterilisation. The effect is largely due to the nascent chlorine liberated, and in some degree to the ozone and hypochlorites formed.
B. W. ANDERSON.

Alloy resistant to sea-water. STERNER-RAINER.—See X.

PATENTS.

Treatment of residues obtained from house refuse. G. H. HADFIELD (E.P. 272,981, 13.3.26. Cf. E.P. 249,247; B., 1926, 428).—The wet mixture of coal, coke, and vegetable matter, obtained from house refuse by wet-separating processes, is heated so that the vegetable matter is dried to the point of charring or is actually charred or burnt. F. R. ENNOS.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPTEMBER 9, 1927.

I.—GENERAL; PLANT; MACHINERY.

Ball and tube flowmeter suitable for pressure circuits. J. H. Awbery and E. Griffiths (Proc. Physical Soc., 1927, 39, 296—299).—A robust form of the Ewing ball-and-tube flowmeter employing an inclined tube and suitable for the metering of fluids under pressure, *e.g.*, ammonia in a refrigerating plant, is described. The behaviour of the instrument under conditions of pulsating flow is examined.

J. S. G. Thomas.

Carbon crucible for very high temperatures. M. K. Hoffmann (Z. Elektrochem., 1927, 33, 200—202). —A carbon crucible is dried at 150° and then soaked for 1 hr. at ordinary temperature in a 1·5% sol of silica or vanadium pentoxide. This process is repeated several times. On heating at high temperatures the corresponding carbide is produced and the durability of the crucible is thereby greatly increased. The outside of the crucible may be painted with "Thermonit," a preparation of alumina. The process is compared with that of Ruff and Konschak (A., 1927, 102). H. J. T. Ellingham.

Contamination of samples ground in iron mortars. O. Hackl (Chem.-Ztg., 1927, 51, 442).— After grinding a sample of pure borax glass in an iron mortar it contained 0·79% Fe; the same sample after treatment with a magnet still contained 0·11% Fe. To avoid contamination of samples in this way coarse crushing in a stone rock-breaker, followed by fine crushing by pounding in a "diamond" steel mortar, is recommended. Very fine grinding should be done in an agate mortar. A. R. Powell.

Patents.

Separation of liquids and solids of low melting point by centrifuging. Bergedorfer Eisenwerk A.-G. (G.P. 437,482, 4.9.23. Conv., 4.9.22).—The solid of low m.p. is heated and separated in a viscous or liquid state without interrupting the operation of the centrifuge. The heating is carried out conveniently by the addition of a quantity of material of the same composition as the solid to be separated, which has been heated above its m.p. A. B. Manning.

Condensation of vapours and extraction of non-condensable gases. R. Mercier (E.P. 265,199, 26.1.27. Conv., 26.1.26).—The gas and a liquid are passed together through a centrifugal rotor which is provided with two kinds of liquid jets; one set of small area and/or larger radius produces a high velocity to transport the gas, and the other jets have larger area and lower velocity to produce a larger volume of spray to scrub the gas. B. M. Venables.

Apparatus for cooling by the expansion of gases. V. Cremieu (U.S.P. 1,625,712, 19.4.27. Appl., 9.1.26. Conv., 12.5.25).—The gas is expanded through narrow passages formed through and along a tapered seat on the end of the supply pipe. The first tapered seat is surrounded by another attached to a tube surrounding the supply tube, and having slight freedom of movement. Outwardly again are another fixed tube and a final movable tube. B. M. Venables.

Apparatus for condensing vapours. E. W. Isom and J. E. Bell, Assees. to Sinclair Refining Co. (U.S.P. 1,627,544, 3.5.27. Appl., 23.6.21).—A number of condensing tanks arranged in series are each arranged to contain both vapour and liquid condensate, and are each provided with a cooling coil. In any tank, vapour taken from the one before (or from the supply) is sprayed into the mass of already-formed condensates. The several condensates are drawn off separately.

B. M. Venables.

Sub-cooling condenser. R. J. Pabodie, Assr. to Griscom-Russell Co. (U.S.P. 1,625,821, 26.4.27. Appl., 5.5.21).—The vapour and condensate pass through tubes arranged in a number of sections, the total cross-sectional area of the tubes being constant in each section, but varying section by section inversely as the density of the vapour–liquid medium.

B. M. Venables.

Machinery for separating particles from air by centrifugal action [cyclone separators]. T. Robinson & Son, Ltd., C. J. Robinson, and W. N. Robinson (E.P. 267,580, 19.11.25).—Two cyclone separators are operated in tandem with a fan between them, so that the first operates under partial vacuum and the second under positive pressure. They are each of cylindro-conical construction and of the same diameter, but the cylindrical part of the second is deeper. The central outlet pipes are carried down inside to below the cylindrical part, and the lower end of these pipes is perforated (in addition to the open end) preferably by cutting and raising portions so as to act as collecting scoops for the air rotating in the central part of the vortex. B. M. Venables.

Apparatus for cleansing air and gases. E. C. R. Marks. From De Vilbiss Co. (E.P. 268,163, 3.5.26).— An arrangement of discs and baffles within a casing to effect, *e.g.*, the removal of oil from compressed air.

B. M. Venables.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Reactivity of coke. S. W. Parr and W. D. Staley (Ind. Eng. Chem., 1927, **19**, 820—822).—The "ignition temperature" of coke, *i.e.*, the temperature at which it commences to react with oxygen, rises with the coking temperature. Cokes from an Illinois coal carbonised at temperatures ranging from 500° to 1000° showed a progressive rise in ignition temperature from 144° to above 600°. This variation cannot be explained by graphitisation. Amorphous carbon prepared from sugar by the action of sulphuric acid shows a similar progressive fall in reactivity after being heated at successively higher temperatures. It is concluded that for each advance in temperature carbon assumes a definite form which directly affects its reactivity. A. B. Manning.

Uniform heating of coke ovens. H. Kuhn (Fuel, 1927, **6**, 267—277, 318—331).—With the increased height of modern coke ovens the older systems of heating lead to excessive vertical temperature gradients in the oven. The longer time then required to complete the coking of the upper part of the charge, and the consequent overheating of the lower part, have serious disadvantages, *e.g.*, increased heat consumption, non-uniformity of the coke, lower yield of by-products, etc. The advantages and disadvantages of modern heating systems are summarised. Those based on single-flame heating are unsatisfactory. Multiflame systems, in which the air is admitted in several stages from the sole flue upwards, *e.g.*, that used in the Still oven, solve the problem of uniform heating without introducing any new defects. The coking period is about 20% less, the heat consumption about 30% less, and the yields of tar, ammonia, and benzene correspondingly higher, than in a similar single-flame heated oven. The system permits of easy regulation; it has been applied with success to ovens 4 m. high, and a further increase of height to 5 or 6 m. is probable in future designs. A. B. Manning.

Modern carbonising economics exemplified by results and working costs at Portsmouth Gas Works. T. Carmichael (Gas World, 1927, **86**, 604—622).—Detailed results and working costs obtained from tests carried out on different types of gas-making plant at Portsmouth Gas Works are given. The following figures are the minimum costs of gas production per therm for each type of plant, excluding capital and maintenance charges, and allowing credit for the residual products and for the steam produced from the waste-heat boilers with which each type of plant is equipped:—vertical retorts (Durham coal), 1·25d.; horizontal retorts (Durham coal), 1·34d.; blue water-gas, 2·44d.; carburetted water-gas, 3·60d. Economy in working is claimed by screening the coal and using the fines in horizontal retorts and the screened coal in verticals. Information is given regarding the behaviour of the refractory materials used in the construction of the vertical retorts. H. D. Greenwood.

Ovens as a gasworks carbonising plant. G. M. Gill (Gas World, 1927, **86**, 633—646).—A survey of the different types of carbonising ovens which might be adapted for use in gas works, typical results and comparative economics of each type being discussed. It is suggested that in the case of small works a horizontal oven with a coal capacity of 1—2 tons/12 hrs. might replace horizontals, whilst a slightly larger oven unit carbonising 4—6 tons/12 hrs. might be adapted for use in works of medium size. In the case of larger works a choice is available between horizontal, inclined, or vertical intermittent ovens, the latter two types being favoured on the Continent. It is considered that the coke from these ovens is very suitable for central heating, and the rapid development of the coke boiler should remove one of the main objections to the use of large carbonising units. H. D. Greenwood.

Cleaning of blast-furnaces gases. Kling–Weidlein gas cleaner. G. W. Hewson (Fuel Econ., 1927, **2**, 251—253).—Some details and working results are given for two Kling–Weidlein gas-cleaning plants, each capable of dealing with the output of gas from one blast furnace (cf. B., 1924, 209). In one 5 hr. test the dust content of the gas was reduced from 1·03 to 0·184 grain/cub. ft., and the average drop in temperature of the gas on passing through the apparatus was 50—60°. The power required to operate the cleaner and cost of cleaning the gas are indicated. A. B. Manning.

Modern design of carbon dioxide recorders and indicators. A. Grounds (Fuel Econ., 1927, **2**, 233—235).—A brief account is given of three modern types of carbon dioxide recorder, the Cambridge Instrument Co.'s (cf. Engineering, 1925, **119**, 53), the "Ranarex" (cf. Power User, 1922, 302), and the "Union" (cf. B., 1922, 235 A; 1924, 814), to illustrate the improvement in design due to utilising the physical properties of gases in such instruments. A. B. Manning.

Coking of oil shales. W. L. Finley and A. D. Bauer (U.S. Bur. Mines, Tech. Paper, 1926, No. 398, 11 pp.).—In order to examine the progress of the coking exhibited by some shales, a coking shale was heated in a standard assay retort until 10% of the oil had been produced; the retort was then cooled and opened. In the upper part of the retort the shale was unchanged, the bottom of the retort held dense, hard coke, and in between was a layer of semi-plastic shale. The shale apparently melts below its temperature of decomposition. By mixing a non-coking shale with a coking variety, satisfactory removal of the spent shale from the retort was accomplished. Thus, addition of 40% of a non-coking shale produced a distinctly softer coke than when a coking shale alone was used, whilst a mixture of 20% of coking with 80% of non-coking shale gave a spent shale which stuck together, but which could be easily broken apart. Mixing of a coking shale with a spent shale produced somewhat similar results as regards the resulting spent shale. The effect of steam was examined; external heat raised the temperature of the shale to 250°, saturated steam was then blown in, and the temperature gradually raised to 555°. A coke was obtained neither so hard nor so dense as when no steam was used. Steam at 650° blown through the shale without application of external heat produced coke just as hard and dense as when external heat alone is

applied. After 100 hrs. roasting at 165—186° practically no oil was obtained on retorting. Preliminary oxidation at 125—135° destroyed the coking properties of a shale, increased both the amount of water produced and the fixed carbon in the spent shale, and decreased the oil yield by nearly 55%. The oil so produced had a higher sp. gr. and a lower setting point than the oil from raw shale, and yielded on distillation a more unsaturated distillate giving a highly carbonaceous residue. Extraction of powdered shales with carbon tetrachloride, pyridine, or xylene gave no indication of the coking properties of the shale. Extraction with pyridine removes some of the coking constituents from the shale, but the amount of extract cannot be determined accurately owing to the interaction of the pyridine with organic matter in the shale. W. N. HOYTE.

Composition of petroleum (kerosene and other) fractions, with the standardisation of miscibility and optical tests. J. A. CARPENTER (J. Inst. Pet. Tech., 1926, **12**, 518—567).—Solubility of aniline in hydrocarbons is greatly affected by the presence of water, and yields useful information as to the composition of hydrocarbon fractions. The research concerns the correlation of this method with optical and other methods; the study of the liquid systems resulting from aniline and *o*-toluidine with hydrocarbons or water; the separation of hydrocarbons in petroleum fractions; and the study of fractions above 300°. Details are given of the examination of fractions of Burmah crude, American kerosene, and Badarpur crude fractions. The nature of the unstable compounds in kerosene fractions is discussed, and an attempt made to identify them. In determining unsaturateds and aromatics the losses of hydrocarbon on acid treatment are much less accurate than the aniline point determinations. H. MOORE.

Burkheiser ammonium sulphite-bisulphite process. TERRES and HAHN.—See VII.

PATENTS.

Dust removal from carbonisation gases containing tar vapours. KOHLENSCHEIDUNGS-GES.M.B.H. (G.P. 439,251, 2.12.21).—The gases pass through a filter which can be heated at a temperature above the dew-point of the highest-boiling constituent of the tar vapours. Or, by working at a lower temperature, the dust, together with some of the higher-boiling constituents of the tar, is deposited on the filter mass, which consists of gas-coke, semi-coke, or other distillation residues, in such a manner that the latter can be briquetted without further addition. A. B. MANNING.

Conversion of heavy into lighter hydrocarbons. DEUTSCHE ERDÖL A.-G. (E.P. 244,107, 3.12.25. Conv., 3.12.24).—Oil is preheated under pressure to the temperature of its conversion into lighter hydrocarbons and then transferred to a reaction chamber, the temperature of which can be controlled independently of the preheaters, in which the reaction is completed. The product is discharged into a low-pressure rectifying column from which the desired light hydrocarbons pass as vapours and are condensed, whilst the heavier fractions are returned to dilute the hydrocarbons to be treated. S. PEXTON.

Separating a mixture of a large number of volatile liquid and gaseous hydrocarbons. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ, and J. H. C. DE BREY (E.P. 255,428, 3.6.26. Conv., 14.7.25).—Casing-head gasoline, rich in constituents volatile at ordinary temperatures and pressures, is fed to the middle of the rectifying column of a still which operates under 4 atm. pressure. The boiling and the reflux are regulated so that the top fraction, which usually amounts to 20% of the crude gasoline, has a boiling range of 0° to below 100°. This fraction is discharged in the gaseous condition, and can be used as fuel. The bottom fraction, of boiling range 0—150°, flows under its own pressure to the middle of a fractionating column working under 2 atm. pressure, where it is divided into fractions boiling at 0—30° and 30° to above 150°, respectively. The first fraction is suitable as a fuel for blending with heavy gasoline, the other is run to a fractionating column working at 1½ atm. pressure where it is resolved into a stable motor fuel boiling at 30—100° together with a heavy fraction which can be blended with the more highly volatile fractions. S. PEXTON.

Treating hydrocarbons. W. R. HOWARD, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,627,436, 3.5.27. Appl., 9.6.22. Renewed, 3.3.27).—Oil is vaporised and the vapours are passed through a supply of oil contained in a closed shell within the still. The uncondensed vapours are subjected to reflux condensation, the condensate being returned to and re-treated in the shell. The operation is carried out under pressure. C. O. HARVEY.

Treatment of hydrocarbons. O. G. MESSENGER, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,623,061, 5.4.27. Appl., 6.1.21).—Refinery waste oils are vaporised and passed at low pressure through a cracking tube at about 400° into which chlorine is introduced. Low-boiling chlorohydrocarbons of value as solvents are obtained. T. S. WHEELER.

Condensing, treating, and washing hydrocarbon vapours. C. L. FREELAND (U.S.P. 1,627,431, 3.5.27. Appl., 9.9.21).—The vapours are passed through the hollow passageway formed in a volume of water by spraying it into a tube in such a manner as to impart a whirling spiral movement. C. O. HARVEY.

Treating petroleum and other hydrocarbons. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,627,159, 3.5.27. Appl., 30.11.17. Renewed 16.7.23).—The oil is cracked under pressure by passage through a series of chambers maintained at progressively increasing temperatures, the oil supply to the chamber of lowest temperature being continuous and the vapours from this chamber being condensed and collected. The condensable vapours from the other chambers are returned thereto by means of reflux condensers, and the remaining vapours are forced back through the pool of oil contained in the chamber of next lower temperature. Uncondensable gases formed are collected and passed through the oil. C. O. HARVEY.

Treating oil shale. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,627,162—3, 3.5.27. Appl.,

10.1.21. Renewed, 4.2.27).—(A) The crushed shale is heated in a vessel consisting of an upper and a lower drum connected by a number of vertical tubes heated externally, and also by means of superheated steam in actual contact with the shale, which is fed to the upper drum and passes downwards through the tubes. The evolved vapours are condensed. (B) Aluminous shale is destructively distilled under pressure in a similar apparatus, and chlorine is passed through the hot shale in countercurrent to its direction of movement.

C. O. HARVEY.

Cracking hydrocarbon oils. CHEMICAL RESEARCH SYNDICATE, LTD., Assees. of J. B. WEAVER (E.P. 253,562, 15.6.26. Conv., 15.6.25).—See U.S.P. 1,601,786; B., 1927, 6.

Cracking of hydrocarbons. E. C. HERTHEL and H. L. PELZER, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,634,666, 5.7.27. Appl., 8.4.24).—See E.P. 232,178; B., 1925, 907.

Apparatus for removing [coke] deposits from stills and the like. A. E. WHITE. From TEXAS Co. (E.P. 273,159, 29.11.26).

Process and apparatus for the gasification of solid fuels. L. CHAVANNE (E.P. 247,571, 10.2.26. Conv., 11.2.25).

Coke-extracting mechanism for vertical retorts for the distillation of carbonaceous materials. F. J. WEST, E. WEST, and WEST'S GAS IMPROVEMENT Co., LTD. (E.P. 272,691, 28.6.26).

[Charging device for use in the] low-temperature distillation of fuel. J. PLASSMANN (E.P. 260,569, 11.10.26. Conv., 27.10.25).

Apparatus for carburetting water-gas. [Automatic] apparatus for making water-gas and other cyclical gas-making processes. HUMPHREYS & GLASGOW, LTD., Assees. of H. G. TERZIAN (E.P. 266,680—1, 24.12.26. Conv., 1.3.26).

Apparatus for removing and quenching hot coke discharged from retorts or furnaces. H. J. TOOGOOD, and R. DEMPSTER & SONS, LTD. (E.P. 264,013, 13.1.26).

Manufacture of air gas from inflammable volatile liquids. H. FOERSTERLING (E.P. 248,770, 5.3.26. Conv., 5.3.25).

IV.—DYESTUFFS AND INTERMEDIATES.

PATENTS.

Manufacture of azo dyes containing metal. SOC. CHEM. IND. IN BASLE (GES. FÜR CHEM. IND. IN BASEL) (E.P. 249,884, 26.3.26. Conv., 28.3.25).—A sulphonated 2-diazo-1-naphthol-8-sulphonic acid is coupled with any second component and the product boiled with a chromium or copper compound. Further, 1-naphthol-8-sulphonic acid is coupled with an *o*-diazophenol and boiled with a chromium compound, or with an *o*-diazonaphthol and boiled with a copper compound. From 2-amino-1-naphthol-4:8 (or 3:8)-disulphonic acid, diazotised and coupled with 1-phenyl-3-methyl-5-pyrazolone, the copper compound dyes wool red, the chromium compound blue-red. The same diazo compound is coupled with 1-naphthol-4:8-disulphonic acid (copper, violet), with α- or β-naphthol (chromium, navy blue), (with 2:3-hydroxynaphthoic or 2-naphthol-4:5-dicarboxylic acid (chromium, blue), with resorcylic acid (chromium, violet), with 1-naphthol-3:8-disulphonic acid (chromium, blue). 1-Naphthol-4:8(or-3:8)-disulphonic acid is coupled with diazotised 4-nitro-2-aminophenol (chromium, bordeaux), with diazotised 4-chloro-2-aminophenol (chromium, red-blue or violet), with diazotised picramic acid (chromium, black). If carboxyl groups are present, the dye is suitable for chrome printing on cotton.

C. HOLLINS.

Manufacture of anthraquinone derivatives. BADISCHE ANILIN U. SODA-FABR., Assees. of FARBENFABR. VORM. F. BAYER & Co. (E.P. 244,462, 8.12.25. Conv., 19.12.24).—The labile compounds from 4-substituted 1-aminoanthraquinones and formaldehyde condensed as described in G.P. 123,745 (A., 1902, i, 119) are further condensed in acid media (especially 65—85% sulphuric acid) to give new products of unknown constitution. The condensation is facilitated in certain cases by addition of boric acid. Two series of products, *a* and *b*, are formed according as 1 mol. or 2 mols. (or more) of formaldehyde react. Moderated oxidation of the *b*-compounds gives further new products, from which formaldehyde may be split off, with formation of *a*-compounds; further oxidation yields the products of E.P. 244,463 (cf. following abstract). To 1-amino-4-hydroxyanthraquinone sulphuric acid, heated at 90° and cooled to 25°, paraformaldehyde is added and the bluish-violet solution of the intermediate product is kept for 12—36 hrs. at 15° or is heated at 70—75° for a short time. The colour changes to green and a product of the *b*-type separates. With less paraformaldehyde at 40° the *a*-product is obtained. A mixture of *a*- and *b*-products gives blue-grey shades on cotton from a hyposulphite vat. Similar products result when 1:4-diaminoanthraquinone is used. 4-Amino-1-methoxyanthraquinone gives a bluish-grey to black vat dye.

C. HOLLINS.

Manufacture of anthraquinone derivatives. BADISCHE ANILIN U. SODA FABR., Assees. of FARBENFABR. VORM. F. BAYER & Co. (E.P. 244,463, 8.12.25. Conv., 9.12.24).—α-Aminoanthraquinones are condensed with formaldehyde as described in E.P. 244,462 (cf. preceding abstract) to give compounds of series *a* or *b*, which are then oxidised (more energetically than in the earlier patent), forming new quinone-like vat dyes. For the oxidation, manganese dioxide and sulphuric acid are used. From 1-amino-4-hydroxyanthraquinone a greenish-blue vat dye is obtained.

C. HOLLINS.

Violet vat dye of the 1-thionaphthen-2-indole-indigo series. R. HERZ, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,631,865, 7.6.27. Appl., 5.8.26. Conv., 6.11.24).—The process of U.S.P. 1,590,685 (B., 1926, 816) is modified in that 3-alkyl derivatives of the hydroxythionaphthen compounds therein claimed are employed. *E.g.*, 4:6-*dichloro-2-hydroxy-3-methylthionaphthen*, m.p. 135°, from 4:6-dichloro-*m*-toluidine through 4:6-*dichloro*-m-*tolylthioglycollic acid*, m.p. 106°, using the methods of the patent cited, is condensed with 2:5:7-trichloroisatin to give a *dye*, yielding on cotton and wool

in a hyposulphite vat, bluish-violet shades fast to washing, light, and chlorine. T. S. Wheeler.

Manufacture of water-soluble condensation products. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 251,294, 23.4.26. Conv., 23.4.25).—An aromatic sulphonic acid or a derivative thereof other than a hydroxy-sulphonic acid is condensed with an aromatic hydroxy-alcohol, a derivative thereof, an aliphatic, aromatic, or hydroaromatic alcohol, a phenol, or a compound containing a replaceable halogen atom. In place of the aromatic sulphonic acid may be used aldehyde or other condensation products of an aromatic sulphonic acid of high mol. wt., or an aromatic hydrocarbon with subsequent sulphonation. Condensation occurs at moderate temperatures, and, if desired, in the presence of mineral acid. S. S. Woolf.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Viscose. XI. G. Kita, R. Tomihisa, K. Sakurada, Y. Nakamura, and H. Kono (J. Cellulose Inst., Tokyo, 1927, **3**, 117—125).—Viscose containing 6% of cellulose was spun into a coagulating bath containing 17·5% of ammonium sulphate, 8% of sulphuric acid, and 7·5% of dextrose, the influence of the time of ageing of the alkali-cellulose and ripening of the viscose and the alkalinity of the viscose being studied. The viscose from alkali-cellulose aged only for a short time spins more easily under a high pressure, whereas that from more seasoned alkali-cellulose spins under a lower pressure. The viscose spins more easily as its ripening advances. The ease of spinning is measured by the speed; a speed of 57 m./min. is not easily obtainable when the alkali-cellulose has been aged for too short or too long a time, but is readily obtained with viscose from moderately aged alkali-cellulose. With the same viscose, differences in tenacity and elongation of the thread may be produced by differences in spinning speed and pressure owing to variations in the coagulation conditions, *i.e.*, the velocity of the action of the bath on filaments of different thickness. The tenacity and elongation increase with the times of ageing of the alkali-cellulose and ripening of the viscose up to a certain point, beyond which they remain constant or decrease slightly. It would not be right, however, to conclude that the increase in tenacity and elongation is dependent on the decrease in viscosity of the viscose which takes place at the same time. J. F. Briggs.

Comparing the cleanliness of sulphite pulps. T. L. Crossley (Pulp and Paper Mag., 1927, **25**, 771).—The comparative cleanliness of sulphite pulps is judged from their content of readily-reducible sulphur compounds, as determined by reduction with zinc and hydrochloric acid in a vessel, the outlet of which is covered with a lead acetate paper. 0·25—0·1 g. of the sample is used for the test, and standard stains are made from 0·0001*N*-sodium thiosulphate. By this method bleached pulps usually show from 0·00008% to 0·1% S. D. J. Norman.

Patents.

Production of artificial fibres from viscose. H. Jentgen (G.P. 439,359, 17.1.25).—Ammonium salt solutions, used as the first precipitation bath in the two-bath process, contain aldehydes, with or without sugars, vegetable extracts, etc. L. A. Coles.

Preparation of spinning baths for viscose. Vereinigte Glanzstoff-Fabr. A.-G. (G.P. 438,461, 11.5.19).—Viscose threads on leaving the coagulating bath are washed with dilute sulphuric acid whereby salts are removed as readily soluble bisulphates. The wash liquor is then concentrated, if necessary, and further quantities of sulphuric acid are added. D. J. Norman.

Manufacture of fine viscose filaments. Vereinigte Glanzstoff-Fabr. A.-G. (G.P. 438,460, 24.1.19).—By increasing the concentration of the acid in the coagulating bath the thread becomes progressively finer. D. J. Norman.

[Non-inflammable] cellulose composition. W. G. Lindsay, Assr. to Celluloid Co. (U.S.P. 1,630,752, 31.5.27. Appl., 25.4.22. Cf. U.S.P. 1,233,374; B., 1917, 1004).—The composition comprises pyroxylin (100 pts.), tricresyl phosphate (75 pts.), and hexachloroethane (125 pts.). T. S. Wheeler.

Cellulose acetate composition [film] of low inflammability. S. J. Carroll, Assr. to Eastman Kodak Co. (U.S.P. 1,631,468, 7.6.27. Appl., 27.4.25).—Cellulose acetate film composition is mixed with pure *s*-tribromophenol or similar compound. T. S. Wheeler.

Production of weighted artificial silk. G. Rivat, Assr. to Lyons Piece Dye Works (U.S.P. 1,631,062, 31.5.27. Appl., 15.10.25).—Cellulose acetate silk can be weighted in the same manner as natural silk, if, before treatment with stannic chloride and sodium phosphate solution, it is gelled by soaking in cold 15—20% aqueous formic or acetic acid. T. S. Wheeler.

Manufacture of hollow [artificial silk] fibres. W. O. Snelling (U.S.P. 1,631,071, 31.5.27. Appl., 11.5.25).—Molten sugar is forced through a jet to form a fibre, which is coated with a solution of a cellulose ester, *e.g.*, nitrocellulose or viscose, the soluble core being removed by treatment with water after coagulation and the hollow fibre treated in any usual way, *e.g.*, denitrated. T. S. Wheeler.

Manufacture of paper pulp. J. B. Beveridge (U.S.P. 1,631,789, 7.6.27. Appl., 13.4.25).—Cellulosic material is digested with an aqueous solution containing sodium hydroxide, sodium sulphide, and the waste liquor from a pulping process in which a solution of sodium acid sulphite has been used. D. J. Norman.

Manufacture of a paper product. J. McIntosh, Assr. to Diamond State Fibre Co. (U.S.P. 1,631,750, 7.6.27. Appl., 2.3.26).—A cellulose ester is added to the pulp before it reaches the Fourdrinier machine. The resulting paper is parchmentised, and is then subjected to heat and pressure to cause the cellulose ester to flow and permeate the sheet. D. J. Norman.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Burkheiser ammonium sulphite–bisulphite process. E. Terres and E. Hahn (Gas- u. Wasserfach, 1927, **70**, 309—312, 339—343, 363—367, 389—395).—

The ternary system ammonia–sulphur dioxide–water has been studied from the point of view of the practicability of the Burkheiser process. Solubility curves, showing the variation of the composition of the solution with varying excess of ammonia or of sulphur dioxide, have been plotted for 0°, 20°, 40°, and 60°. They exhibit a number of discontinuities which do not correspond with any definite change in the solid phase; the only solid phases in equilibrium with the solution are NH_4HSO_3 and $(NH_4)_2SO_3, H_2O$, in the region of excess sulphur dioxide, and $(NH_4)_2SO_3,H_2O$, in the region of excess ammonia. The solubility of ammonium sulphite in water rises regularly from 32·9% at 0° to 50·0% at 60°, and that of the bisulphite from 72·8% at 0° to 86·1% at 60°. The partial pressures of ammonia and of sulphur dioxide over solutions of ammonium sulphite are almost linear functions of the concentration; the values for ammonia are much higher than the corresponding values for sulphur dioxide, but both are sufficiently small, even at 40° and over concentrated solutions, to permit a practically quantitative removal of either constituent from a gas by washing. The partial pressures at 20°, 40°, and 60° have also been determined over saturated solutions in the presence of excess of ammonia and of sulphur dioxide. The results definitely demonstrate the possibility of the successful operation of the Burkheiser process, and indicate the most favourable conditions under which it should be carried out.

A. B. Manning.

Commercial sodamide. A. Guntz and F. Benoit (Bull. Soc. chim., 1927, [iv], **41**, 434—438).—Commercial sodamide of dark colour contains metallic iron and up to 16% of sodium hydride; the presence of the latter substance is probably the cause of the accidents sometimes attending the use of sodamide. The action of hydrogen on sodamide and of ammonia on sodium hydride is investigated. In order to prepare pure sodamide the sodium must be heated in a current of ammonia in the region of 300° until it is completely transformed and the greenish liquid decolorised; the melt is then poured on to a cold surface.

S. K. Tweedy.

Calcium sulphate. II. Supersaturated solutions of calcium sulphate, pure, and mixed with other salts. L. Chassevent (Ann. Chim., 1926, [x], **6**, 313—351; cf. A., 1926, 1217).—The effects of changes of temperature and of the addition of varying quantities of the di- and hemi-hydrates on the velocity of crystallisation of saturated solutions of the hemihydrate (prepared from calcium sulphate dehydrated below 300°), which are, therefore, supersaturated with regard to gypsum, have been studied by means of conductivity measurements. Even when special precautions are taken to exclude seeds of the dihydrate, the solutions of the hemihydrate crystallise spontaneously after about 26 min. at 16·5°, the period before crystallisation sets in being longer the higher the temperature. Addition of the dihydrate induces crystallisation at all temperatures, the velocity being greater the larger the proportion of dihydrate added. The velocity of crystallisation of solutions becomes very small above 60°, even in the presence of a large quantity of the dihydrate. The effect of a rise in temperature on solutions saturated with respect to the hemihydrate is to cause immediate crystallisation of the dihydrate. The addition either of anhydrous calcium sulphate (dehydrated at 430°, 590°, 900°, or 1000°), or of the hemihydrate, has no effect in the velocity of crystallisation. The influence of salts, bases, and acids on the velocity of hydration of calcium sulphate has been studied calorimetrically. In general, substances which increase the concentration of the sulphate ions in the solution, either by the direct addition of soluble sulphates or by double decomposition with the calcium sulphate, decrease both the period preceding crystallisation and the duration of the hydration. Thus the addition of potassium chloride increases the velocity of hydration, the increase being proportionately greater for small concentrations of potassium chloride than for larger, and at concentrations of less than 18·2 g./litre the total amount of heat evolved is the same, and the separated crystals consist entirely of gypsum, but at higher concentrations the total heat evolved is greater, and the separated crystals have a composition approximately corresponding with $2KCl,CaSO_4,0{\cdot}5H_2O$. A similar effect is obtained by adding potassium sulphate, only gypsum separating up to a concentration of 24 g./litre. Aluminium sulphate also increases the velocity, only gypsum separating up to a concentration of 160 g./litre, but its action is much greater on solutions of commercial calcium sulphate containing 3% of lime and calcium carbonate, this being due to the action of the lime on the aluminium sulphate, since the addition of a saturated solution of lime to a solution of pure calcium sulphate decreases the velocity of crystallisation. Alum also increases the velocity of crystallisation. Thus the crystallisation of the gypsum is probably induced by a secondary crystallisation, in the solution, of a compound of the hemihydrate or the gypsum with other salts present (either added or as impurities in a commercial sample). Addition of sodium hydroxide to a solution of pure calcium sulphate in the absence of carbon dioxide decreases the velocity of crystallisation, the decrease being greater the larger the concentration of the base, but, conversely, with a commercial sample of calcium sulphate in air, the velocity of crystallisation is increased by the addition of sodium hydroxide. Sulphuric acid increases the velocity of crystallisation up to a maximum at about 4—6*N*, greater concentrations of acid then causing a decrease; a similar effect is observed with hydrochloric acid. Rohland's view (A., 1904, ii, 33) that an increase in the solubility of the gypsum increases the velocity of setting of the plaster cannot be upheld, because the solubility of gypsum is increased in the presence of potassium chloride but decreased in the presence of potassium and aluminium sulphates, whilst all these substances increase the velocity of hydration of the hemihydrate.

J. W. Baker.

Dehydration of gypsum. R. Stumper (Z. anorg. Chem., 1927, **162**, 127—140).—The heating curve of gypsum shows that the temperature of transition from dihydrate to hemihydrate rises with increase in the rate at which the temperature of the mass is raised. The change from the hemihydrate to the anhydrous salt

takes place in all cases at about 190°. Following the dehydration gravimetrically yields similar results. Unless the rate at which the temperature is raised is 3—25°/min., the first stage in the dehydration may not be observed. R. CUTHILL.

Production of magnesium sulphate from Manchurian magnesite. R. MATSUURA (J. Soc. Chem. Ind. Japan, 1927, **30**, 190—194).—Magnesite is converted into magnesia by light burning, then into magnesium sulphite by the action of sulphur dioxide, and finally into sulphate by means of oxygen in the presence of cobaltous sulphate. Manchurian magnesite used has the following composition: 44·65—47·95% MgO, 0·43—4·13% CaO, 0·09—0·19% Al_2O_3, 0·33—1·14% Fe_2O_3, 0·28—5·50% of matter insoluble in hydrochloric acid, and 49·04—51·60% burning loss. The calcined magnesite yielded magnesia of 91·32% purity. 48 kg. of the product are charged with 270 litres of water into a wooden barrel having a conical bottom. After closing the barrel and converting the oxide into sulphite by passing in sulphur dioxide, 50 g. of cobalt sulphate and 300 litres of water are added, together with sufficient magnesia (about 1·6 kg.) to neutralise the solution, and compressed air is introduced to oxidise the sulphite. After filtration, the solution is evaporated, yielding about 200 kg. of magnesium sulphate crystals. A semi-commercial experiment was successfully carried out with a unit producing 1 ton of pure sulphate crystals from 225 kg. of oxide. K. KASHIMA.

PATENT.

Production of sodium fluoride. E. P. STEVENSON, Assr. to A. D. LITTLE, INC. (U.S.P. 1,634,122, 28.6.27. Appl., 8.10.21. Renewed 1.12.26).—Sodium fluosilicate is decomposed into sodium fluoride and silicic acid by treatment with a carbonate evolving carbon dioxide in the reaction. The silicic acid is dissolved in alkali, separated from the sodium fluoride, and then treated with carbon dioxide to decompose the silicate and form a bicarbonate suitable for conversion into carbonate for use again in the process. W. G. CAREY.

Purification of alkali-metal cyanide solutions containing sulphides. I. G. FARBENIND. A.-G. (E.P. 262,456, 3.12.26. Conv., 4.12.25).—The solution is treated with bismuth hydroxide or a bismuth salt whereby bismuth sulphide is precipitated without any secondary reactions. W. G. CAREY.

Manufacture of stable peroxidised composition. J. A. L. VAN DER LANDE, Assr. to NOVADEL PROCESS CORP. (U.S.P. 1,631,903, 7.6.27. Appl., 3.5.26. Conv., 18.10.24. Cf. E.P. 234,163; B., 1925, 613).—Hygroscopic flour improvers, *e.g.*, ammonium persulphate, are mixed with non-poisonous, anhydrous salts, *e.g.*, calcium sulphate, which can form stable crystals containing water of crystallisation. T. S. WHEELER.

Production of copper oxychloride. MOUNT LYELL MINING & RAILWAY CO., LTD., Assees. of E. W. TREND (Austral. P. 22,518, 1.4.25).—Copper oxychloride precipitated by treating cupric chloride solution with potassium or magnesium carbonate has good adhesive properties and covering power, and is of uniform colour. L. A. COLES.

VIII.—GLASS; CERAMICS.

Measurement of the softening temperature of glasses. J. T. LITTLETON, JUN. (J. Amer. Ceram. Soc., 1927, **10**, 259—263).—The softening temperature, at which the glass possesses a certain definite viscosity, is that temperature at which a thread (22·9 cm. in length and of uniform thickness, 0·65—1·0 mm.) elongates under its own weight at the rate of 1 mm./min. Since both surface tension and sp. gr. affect the force tending to elongate the specimen, such a conception is only approximate. In the experimental determination the thread, which is formed to a needle point and bent at right angles at one end to serve as a pointer, is suspended from the other end in an electric furnace, at 50—100° below the softening point. The temperature is then raised at the rate of 5—10° per min. Measurements of temperature and elongation are taken every 30 sec., and the results reported graphically. From the graph the softening temperature is easily computed. The control data so obtained also serve to indicate differences in the viscosity of glasses of varying composition. A. T. GREEN.

Antimony white enamel. H. MELZER (Keram. Rundsch., 1926, **34**, 801—803; Chem. Zentr., 1927, 1, 785).—The mixture for producing the enamel must be easily fusible as compared with the foundation; the enamel must have the correct coefficient of expansion, high tensile strength, and good turbidity, and must contain not more than 8% Sb_2O_3 and 7% Al_2O_3, whilst the remaining constituents must be carefully selected. L. A. COLES.

Cause of the white ring on the edge of glasses melted by coal gas. G. OFFE (Gas- u. Wasserfach, 1927, **70**, 435—437).—The ring produced when the edges of glassware are heated to soften in the coal-gas flame is due to the formation of sulphurous acid by combustion of organic sulphur compounds in the gas. This partly oxidises to sulphuric acid, which, by union with the lime of the glass forms calcium sulphate, a molten ring of which adheres to the glass. With glasses containing little lime the ring is nearly or quite absent, whilst with lime glasses the effect increases with increase of flame temperature. The trouble was greatly reduced by provision of a simple device which largely destroyed the sulphur compounds in the gas. A. COUSEN.

Effect of various sodium silicates and other electrolytes on clay slips. S. J. McDOWELL (J. Amer. Ceram. Soc., 1927, **10**, 225—237).—Commercial sodium silicate varies widely in chemical composition, constitution, and physical properties. One variety may have two and a half times as much silica as another, consequently differences in the deflocculating effect of different varieties may be anticipated. Six clays, including china clays and ball clays, were sampled. A portion of each was dried for 24 hrs. at 70° after having been passed through 8-mesh, and made into a slip with twice its weight of water. The slips were well shaken in a ball-mill and passed through 65-mesh, after which they were placed in sealed jars. The sp. gr. of the slips was 1·25 ±0·01. Controlled quantities of the different silicates (increments of 0·005% Na_2O based on the weight of the clay in the slip) were added, and (*a*) the

rate of flow through an "efflux" viscosimeter; (*b*) the p_H value by the method of Randolph and Donnenwirth (B., 1926, 823); and (*c*) the amount of settling over a period of 16 hrs. determined. The results are reported graphically. In all cases of silicate additions a maximum rate of flow is produced when the slip is acid, and those silicates high in silica have the most pronounced deflocculating effect for a given Na_2O content. The p_H value at which clay particles attain maximum charge is lower for sodium silicate solutions than for sodium hydroxide or carbonate solutions. In this connexion the influence of colloidal silica is discussed. The use of sodium silicate rich in silica in preference to sodium carbonate seems desirable where soda blisters are a difficulty. Possible explanations of the mechanism of deflocculation are put forward. A. T. Green.

Particle size distribution of typical felspars and flints. E. Schramm and E. W. Scripture, jun. (J. Amer. Ceram. Soc., 1927, **10**, 264—267).—The results of tests on the size of the particles of felspars and flints, ground by the continuous tube mill, the batch mill, and the Hardinge mill with air separator, are presented. The method of testing has been previously reported (B., 1925, 500). The data show the misleading character of screen tests and indicate the advantage of carrying the analysis beyond the range of screens. Whilst the continuous systems with air separation are most efficient in producing material to pass the screens, the batch-ground product of the same screen test has the smaller average size. A. T. Green.

Glazes of low fusibility containing rutile. M. M. French and C. M. Harder (J. Amer. Ceram. Soc., 1927, **10**, 268—269).—A very fusible lead glaze containing rutile was used as the base, to which oxides of cobalt, copper, manganese, uranium, and iron were added as colorants. When the amount of rutile was increased, felspar was added to check the fluidity. A number of glazes were formed from this base, giving at cone 09—08 very good matts in dull mottled colours. At cone 04—03 the rutile crystals formed in stripes, the colour depending on the oxide used. These glazes give an interesting variety of colours and textures, and will fit a "Monmouth Clay" body modified with 10—20% of flint. A. T. Green.

IX.—BUILDING MATERIALS.

Patents.

Wood preservative. K. H. Wolman, F. Peters, and H. Pflug (U.S.P. 1,622,751, 29.3.27. Appl., 30.1.26. Conv., 2.1.26).—Complex salts of tartaric acid with arsenic and an organic base, *e.g.*, aniline or quinoline, are dissolved in a mineral oil, and used to impregnate wood. T. S. Wheeler.

Wood preservative. Stockholms Superfosfat Fabr. Aktiebolaget (Danish P. 34,370, 28.11.23).—The phenols and phenol ethers used in the preservative described in E.P. 190,074 (B., 1923, 271 A) may be partly replaced by sulphonic acids or chlorine substitution products. Formaldehyde or paraformaldehyde may also be added to diminish still further any loss of preservative by washing out; this addition, moreover, increases the antiseptic action of the preservative. A typical mixture contains, *e.g.*, sodium fluoride 67%, phenols or phenol ethers 15%, phenol sulphonic acids 10%, and paraformaldehyde 8%. A. B. Manning.

Impregnating wood. A. Dessemond (G.P. 437,131, 29.12.25. Conv., 5.3.25).—After each impregnation the wood is subjected to a partial vacuum at a pressure and for a length of time determined by measurement of the liquid extracted, in order to determine the quantity which finally remains in the wood. The autoclave used for the purpose is connected with a graduated container, in which the liquid removed under reduced pressure from the impregnated wood can be measured, and from which it can then be transferred into the main tank containing the impregnating liquid. The distribution of the liquid remaining in the wood is quite homogeneous. A. B. Manning.

Manufacture of acid-proof cementing compositions. I. G. Farbenind. A.-G. (E.P. 267,396, 13.8.26. Conv., 25.3.26. Addn. to E.P. 256,258; B., 1927, 333).—A silicofluoride is substituted for silica as a cement powder in the prior patent. B. W. Clarke.

Manufacture of cement. T. Rigby (U.S.P. 1,628,000, 10.5.27. Appl., 28.11.25. Conv., 28.7.24).—See E.P. 243,410; B., 1926, 129.

Treatment of wet raw materials in the manufacture of cement. J. S. Fasting, Assr. to F. L. Smidth & Co. (U.S.P. 1,627,553, 10.5.27. Appl., 9.4.25).—See E.P. 227,977; B., 1925, 232.

Treatment of cement raw materials in rotary kilns. M. Vogel-Jorgensen, Assr. to F. L. Smidth & Co. (U.S.P. 1,627,585, 10.5.27. Appl., 25.4.25. Conv., 10.5.24).—See E.P. 255,569; B., 1926, 825.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Theory of overheating phenomena in grey cast iron. H. Hanemann (Stahl u. Eisen, 1927, **47**, 693—695).—On the assumption that the solidification of grey cast-iron takes place according to the equilibria of the iron–graphite system, and that no change takes place in the amount of combined carbon present during further cooling to the ordinary temperature, it is shown that there must be a stable saturation limit for the combined carbon dependent on the total carbon content of the metal; this limit is depressed by a rise in the silicon content. If the amount of combined carbon exceeds this limit the solidification takes place partly according to the iron–iron carbide equilibria, and if it is below the limit solidification ensues wholly according to the graphite–iron system. In the latter case true equilibrium is obtained only if the metal is so far overheated that the graphite is entirely dissolved in the molten iron, and if subsequent cooling is sufficiently slow to allow the separation of completely saturated mixed crystals to take place. The higher the temperature to which the metal is heated the finer is the graphite that is precipitated during cooling at normal rates. Hence there are two opposed reactions taking place during overheating, one accompanied by a decrease in the amount of combined carbon owing to decomposition of the solid solution induced by the fine dispersion of graphite, and the other accompanied by an increase in

the combined carbon by reason of the closer approach of the system to equilibrium. This accounts for the increase followed by a decrease in the combined carbon during overheating of grey cast iron without assuming any change in the molecular condition of the liquid metal, as suggested by Pivovarsky (B., 1925, 805).

A. R. Powell.

Influence of the blast-furnace conditions on the total carbon content of pig iron. A. Michel (Stahl u. Eisen, 1927, **47**, 696—698).—Tests on a number of blast furnaces smelting different classes of iron ores have shown that the carbon content of the pig iron produced with any given charge is dependent solely on the temperature in the furnace, being smaller the lower the temperature. Hence a very basic slag requiring a high temperature for melting results in a high-carbon iron. A low-carbon content is also obtained by the use of a higher ratio of ore to coke, *e.g.*, in the so-called "cold" blast-furnace process.

A. R. Powell.

Properties of high-silicon constructional steel. E. H. Schulz and H. Buchholtz (Giessereiztg., 1926, **23**, 615—622; Chem. Zentr., 1927, I, 644).—Addition of 1% Si to mild steel for rolling increases its elastic limit considerably, but its effect on the ultimate strength is dependent on the other constituents of the steel and on the degree of rolling. The steel must be annealed at a higher temperature than a similar plain carbon steel owing to the effect of silicon in displacing the upper transformation point.

A. R. Powell.

Importance of cementite. R. G. Guthrie (Trans. Amer. Soc. Steel Treat., 1927, **11**, 341—354).—Microexamination of a 0·09—0·15% carbon steel billet for wire-drawing showed that the core contained an appreciable amount of pearlite, whilst the rim showed a large amount of cementite spheroids. The rim showed greater hardness, higher tensile strength, less ductility, less resistance to corrosion, and more rapid etching. In low carbon steels not subject to hardening it is desirable to have the cementite in the spheroidal condition, in which they are more stable, take a more uniform electroplated coating, and are more readily weldable. Photomicrographs are given which show that in high-carbon steels, however, the cementite should be in the lamellar form, which is more readily soluble at the hardening temperature, and the steels give a higher degree of hardness and greater homogeneity.

T. H. Burnham.

Corrosion-fatigue of metals as affected by chemical composition, heat treatment, and cold working. J. D. McAdam (Trans. Amer. Soc. Steel Treat., 1927, **11**, 355—390).—Stress-cycle endurance curves are given for a series of 26 steels, tested in air, in fresh water, and for interrupted exposure to corrosion. The corrosion-fatigue limit may be less than one third of the endurance limit. Specimens that failed in water showed corrosion-fatigue spots with transverse cracks passing through non-metallic inclusions. Spots were not, however, visible on stainless iron unless tested considerably above the corrosion-fatigue limit. For steels containing up to 0·24% C the endurance limit was greater in the quenched and tempered than in the annealed condition. Above 0·36% C the reverse held good, as also for nickel steels. Decrease in the cycle frequency decreased the number of cycles to fracture, but the total time of the test increased. For high-chromium steels the corrosion-fatigue limit was higher than for carbon and nickel steels in both fresh and salt water, and rose with increasing chromium content. Heat treatment had little effect. In salt water the limits were considerably lower than for fresh water. The corrosion-fatigue limits of nickel-chromium steels were higher still. High chromium content was more effective than high nickel content in both fresh and salt water. Increase in corrosion resistance caused increase in corrosion-fatigue limit, but though the effects of variation in tensile strength and ductility were insignificant, there appeared to be an undetermined strength factor. There were slight indications that it might be the notch-toughness of the steel.

T. H. Burnham.

Decomposition of austenite in liquid oxygen. O. E. Harder and R. L. Dowdell (Trans. Amer. Soc. Steel Treat., 1927, **11**, 391—398).—The scleroscope hardness of six austenitic steels after immersion in liquid oxygen increased as follows:—Cobalt-chromium steel, 80·7%; high carbon-chromium steel, 65·3%; 22% nickel steel, 34·4%; hyper-eutectoid carbon steel, 26·1%; high-speed steel, 5·2%; manganese steel, 0%. A typical martensitic structure was developed except in the case of the nickel steel and manganese steel, which showed little or no change. Decomposition of austenite was accompanied by decrease in sp. gr., which was greatest in those steels which showed the highest increase in hardness.

T. H. Burnham.

Transformation of retained austenite into martensite by stress. K. Honda and K. Iwase (Trans. Amer. Soc. Steel Treat., 1927, **11**, 399—410).—On hammering steel rods containing 1·4% C and 5% Cr quenched from 1100° in oil, the intensity of magnetisation increased with the strokes. The transformation of austenite into martensite was probably due to the impulsive shock resulting in an atomic agitation. Rockwell and magnetometric hardness determinations on strips of steel containing 0·84% C and 2·96% Cr showed that in thick specimens a greater amount of martensite was produced by water quenching than by oil quenching, but the reverse in thin specimens, confirming Bain's view that internal stress in quenched austenite steels promotes the martensite transformation. Magnetometric measurements demonstrated that the rise of hardness on immersion of the oil-quenched steels in liquid air increases as the diameter of the specimens decreased.

T. H. Burnham.

Non-corroding steel and iron. A. C. Yorke (Metal Ind., 1926, **29**, 419—420; Chem. Zentr., 1927, I, 174).—A short description of the composition, physical and mechanical properties, and methods of making rustless steel and iron is given. The most favourable temperature for rolling is 1050—1100°, for forging, 900—1150°, and for the subsequent annealing, 750—800°. Forging must be done quickly and, after annealing, cooling must be slow. To remove the oxide film the metal is pickled for 10 min. in 50% commercial hydrochloric acid, then for 25 min. in 20% nitric acid.

A. R. Powell.

Silicon steel as a constructional material and for castings. K. von Kerpely (Zentr. Hütten- u. Walzw., 1926, 30, 411—414, 425—427, 437—440, 455—459; Chem. Zentr., 1927, I, 174).—Silicon steel containing less than 0·04% P has a tensile strength of 51·8 kg./mm.², an elastic limit of 39·8 kg./mm.², an elongation of 26·4%, an impact strength of 8·5 kg./cm.², and a hardness number of 150—155. These properties, combined with its good resistance to repeated impacts and alternating stresses, make the alloy suitable for most types of constructional work in place of the usual steels. The alloy may be welded satisfactorily by the resistance method, but arc welding gives somewhat unsound joints. A. R. Powell.

Location of the carbon atom in boydenite. H. A. Schwatz (Trans. Amer. Soc. Steel Treat., 1927, 11, 277—283).—The author considers that there may be two types of solution of carbon in iron, viz., additive and substitutional, corresponding to austenite and boydenite, the solid solution of the stable system. This is supported by the observation of Osawa that in austenitic steels part of the carbon atoms is substituted for metal atoms. From dilatation data determined in Honda's laboratory the author calculates that the densities of austenite and boydenite at 900° are 7·55 and 7·393 respectively, which difference is of an order consistent with the view expressed. The density of a nickel–iron alloy containing 19·93% Ni and 3·9% of total carbon shows that the solid solution in this case is austenite. T. H. Burnham.

Detection of small inclusions in crystallites with the aid of the production of lines of slip. G. Tammann and H. H. Meyer (Z. Metallk., 1927, 19, 85).—If an indentation is made in a crystallite of a polished surface of metal by means of a gramophone needle the slip lines are regularly oriented around the impression if there are no inclusions in the crystallites. In the presence of such impurities the slip lines are curved round the inclusion, *e.g.*, in meteoric iron, or run towards it, *e.g.*, in copper containing particles of oxide. A. R. Powell.

Metals and alloys for thermocouples for the measurement of high temperatures. W. Rohn (Z. Metallk., 1927, 19, 138—144).—A review of the composition, preparation, working range, and properties of commercial thermocouples. For temperatures above 1000° only alloys of the platinum metals are available, and efficient service is obtained only when metals of the highest degree of purity are used. Traces of calcium, silicon, or carbon derived from the materials used in melting may produce serious errors in the readings, whilst contamination with even 0·02% Fe or Ir produces considerable deviation in the calibration during prolonged use. Of the various base metal couples manufactured for use at temperatures up to 1000° those comprising nickel and chromium with relatively little iron give the most constant readings after prolonged use. Protection tubes of 30% chromium–iron alloy are suitable for use with thermocouples in copper and brass foundries as this alloy remains completely unattacked in molten copper and its alloys. A. R. Powell.

Reduction of mineral oxides. B. Bogitch (Compt. rend., 1927, 184, 883—885).—The solubility of carbon in mineral oxides containing silicates and silico-aluminates of calcium, and small amounts of copper, nickel, and iron, fused rapidly below 1600°, varies from 0·09 to 0·15%, the fused mineral separating from the reducing coke. Hence the difficulty of obtaining complete reduction of a mineral insufficiently reduced at the moment of fusion. The solubility of the carbon increases with temperature, and calcium carbide is formed. The addition of a base (chalk) increases the rate of reduction when fusion occurs rapidly, and the yield of metal rises to a maximum and falls as the amount of base is increased, owing to a diminution of the fluidity of the medium. J. Grant.

Gaseous reduction of tungsten and molybdenum oxides. E. W. Engle (Trans. Amer. Electrochem. Soc., 1927, 51, 345—349).—The reduction of tungsten trioxide by hydrogen commences at 620° and is rapid at 950°, the metal produced by reduction at the latter temperature being most suitable for sintering and subsequent working. The hydrogen consumption per kg. of tungsten powder varies from 900 to 2800 litres. Molybdenum trioxide commences to reduce at 300° in hydrogen, and reduction can be completed at 625°, but it is preferable to carry the reduction nearly to completion at 850°, cool and powder the product, and finish the reduction at 1100°. A. R. Powell.

Gaseous nature of carbon reduction of tin concentrates. C. J. Fink and C. L. Mantell (Trans. Amer. Electrochem. Soc., 1927, 51, 371—381).—Solid carbon does not reduce stannic oxide in an atmosphere of nitrogen below 950°, but, in the presence of a small quantity of air, reduction by graphite commences at about 800° even if the two substances are not mixed together, showing that carbon monoxide is the true reducing agent. A brief description with flow-sheet is given of Bolivian tin-smelting practice. A. R. Powell.

Reduction of tin oxide and cassiterite concentrates. E. F. Kern (Trans. Amer. Electrochem. Soc., 1927, 51, 403—412).—Reduction of stannic and stannous oxides by coal gas starts at about 500°, and is rapid at 700°; by bituminous coal it starts about 600°, and is rapid above 750°, and by charcoal it starts at about 750°, and is rapid above 810°. For the reduction of cassiterite, hydrogen is a much more rapid and efficient reducing agent than carbon monoxide. At a given temperature the percentage reduction by hydrogen–nitrogen mixtures increases with an increase in the hydrogen content, but the efficiency of the reduction decreases; mixtures of nitrogen and carbon monoxide behave similarly. A. R. Powell.

Gaseous reduction of zinc [oxide]. C. J. Maier and O. C. Ralston (Trans. Amer. Electrochem. Soc., 1927, 51, 429—446).—From thermodynamical considerations it is shown that hydrogen and carbon monoxide are almost equally efficient as reducing agents for zinc oxide, and that if carbon monoxide is used it is much more economical to effect the reduction in two stages, (*a*) $ZnO + CO = CO_2 + Zn$, and (*b*) $CO_2 + C = 2CO$, separately. Very rapid reduction of zinc oxide by carbon monoxide takes place at 1200°, but on cooling the gases to condense the zinc vapour, rapid reversion

of the reaction takes place; this could, however, be overcome by subjecting the hot zinc–carbon dioxide mixture to the producer gas-reaction (*b*) directly, but trouble would then be experienced owing to dilution of the zinc vapour and consequent difficulty in effecting condensation. Re-oxidation by water vapour in the case of hydrogen reduction is even more serious than re-oxidation by carbon dioxide. Methane has the advantage over both hydrogen and carbon dioxide that complete reduction of zinc oxide is effected by it below 900°, thus, $ZnO + CH_4 = Zn + CO + 2H_2$, which is not a reversible reaction, but the maximum proportion of zinc vapour in the issuing gases is only 25% by volume using pure methane. A. R. POWELL.

Nickel–silver alloys. C. ROTHERT and G. DERN (Z. Metallk., 1927, **19**, 158—161).—The colour of nickel-silver alloys is governed almost entirely by the nickel content, the whitening effect of zinc being relatively small. Nickel also has a much greater effect on the hardness than zinc. The most satisfactory alloy out of a series of 28 melts of varying composition was found to be that containing 18% Ni, 20% Zn, and 62% Cu, the structure of which resembles α-brass in being a homogeneous solid solution, and in having a pronounced tendency to the formation of twinned crystals on annealing. This alloy, after cold rolling, commences to recrystallise just below 600°, but the best structure is obtained at 800—850°. The work-hardened alloy has a tensile strength of 71·5 kg./mm.^{2} with an elongation of 6·6%; after annealing the corresponding figures are 51 kg./mm.^{2} and 33%. The depth of penetration of an annealed sheet 2 mm. thick in the Erichsen test is 13·0 mm. A high annealing temperature causes a very rapid growth of the crystals with a corresponding reduction in the tensile strength and the danger of the formation of inclusions of zinc oxide along the grain boundaries.
A. R. POWELL.

Influence of molybdenum and silicon on the properties of non-rusting chromium steels. W. OERTEL and K. WÜRTH (Stahl u. Eisen, 1927, **47**, 742—753).—The hardness of plain chromium steels with 14—15% Cr increases rapidly with the carbon content from 0·1 to 0·3% C, then only relatively slowly; in all cases maximum hardness is obtained by annealing at 1050—1100°, above which temperature the hardness begins to fall again, especially in the case of the steel containing 0·5% C. Silicon has practically no effect on the hardness of low-carbon chromium steels, but reduces the hardness of those containing more than 0·3% C. Molybdenum has little effect on the hardness of any of the steels, but it tends to overcome the softening effect of silicon in the higher-carbon steels. Chromium steels containing more than 3% Si do not undergo the A1 and A3 transformations on heating or cooling; they become very coarse-grained during annealing, and have a relatively low tensile strength and tenacity. With up to 1% Si and a low carbon content, chromium steels are characterised by a high Erichsen value, hence they are suitable for the manufacture of hollow vessels. The steel with 0·1% C, 0·32% Mo, and 14—15% Cr becomes very soft and ductile on annealing and has a relatively high elastic limit. Non-rusting steel containing less than 0·3% Mo is highly resistant to corrosion by acetic acid or sea-water, and is more resistant to 5% hydrochloric acid than any other steel so far tested. The silicon steels, on the other hand, are most resistant to nitric acid and to scaling on heating when more than 1% Si is present. After 48 hrs. at 900° the surface of a steel containing 0·15% C, 4·7% Si, and 14·5% Cr was grey and smooth, without any sign of oxide scale.
A. R. POWELL.

Uses of pure nickel. R. J. MCKAY (Trans. Amer. Electrochem. Soc., 1927, **51**, 457—461).—The properties of pure nickel show that the pure metal possesses few commercial advantages over the best samples of nickel which can be produced on the technical scale. The advantages of nickel as a non-corrosive material in engineering design and in food handling processes are pointed out. L. M. CLARK.

Determination of the gas content of molten metals. A. WÜSTER and E. PIVOVARSKY (Stahl u. Eisen, 1927, **47**, 698—702).—An apparatus is described and illustrated by means of which the gas content of metals melted in an electric resistance furnace in a vacuum may be determined, and examples of its use in the examination of the gas content of cast iron poured at varying temperatures are given. In general, the gas content of cast iron increases with the temperature at which the metal is heated before casting up to 1500—1600°, then decreases again; hydrogen is usually the predominant gas, followed by carbon monoxide, carbon dioxide, nitrogen, and methane. A. R. POWELL.

Magnesium and its alloys. W. R. D. JONES (Metal Ind., 1926, **29**, 433—435, 459—461, 463; Chem. Zentr., 1927, I, 645).—Commercial magnesium contains 0·001—0·02% Cu, 0·051—0·01% Si, and 0·059—1·08% Fe, and, in the cast state, has a higher tensile strength than commercial aluminium. Its tensile properties are considerably improved by forging, especially if the castings are free from blowholes, which may be ensured either by adding a small quantity of calcium just before casting or by adopting a special melting process. A magnesium alloy with 8% Al ("Dow metal") has an elastic limit of 1 ton/in.^{2}, a tensile strength of 11·1 tons/in.^{2}, an elongation of 4%, and a Brinell hardness of 55 in the cast state; for the same alloy after forging the corresponding values are 4·6, 21·4, 7%, and 71. The normal oxide film on magnesium and its alloys is an efficient protection against corrosion, as well as an electrical insulator, a film 0·1 mm. thick withstanding 220 volts. The thickness of the film may be increased and its insulating properties improved by heating the metal at 320—330° in an atmosphere of high-pressure steam. A. R. POWELL.

Melting of alloys. M. FOURMENT (Rev. Mét., 1927, **24**, 179—193).—An account of modern electric melting furnace practice.

Protective value of nickel plating. II. C. T. THOMAS and W. BLUM (Trans. Amer. Electrochem. Soc., 1927, **51**, 515—527; cf. B., 1925, 810).—In continuation of earlier work on the behaviour of nickel-plated steel in accelerated corrosion tests, samples of the platings that gave the most satisfactory results in the laboratory were exposed to the atmosphere for 1 year. The behaviour of the specimens showed that the time required to

produce initial corrosion in the accelerated tests was no criterion of the protective value of the coating against atmospheric corrosion. An approximate idea of the relative protective value of nickel coatings is, however, obtained by expressing the porosity of the coating, as determined by the ferricyanide test, in terms of the reciprocal of the number of perforations in a given area. Deposits having a high content of iron turn yellow very quickly on exposure to the atmosphere of a town, and frequently the deposit peels off. The best protection is afforded by nickel deposits having an intermediate copper layer between the nickel and steel. A. R. Powell.

Inductive heating. Northrup.—See XI.

Protective coatings for light-weight alloys. Gardner.—See XIII.

Painting galvanised iron pipes. Gardner.—See XIII.

Patents.

Making fluxing materials.—J. Lund (U.S.P. 1,626,292, 26.4.27. Appl., 23.10.20).—A blast-furnace is operated so as to produce molten iron and a slag which contains an alkali carbonate. Superheated steam is directed on to the surface of the slag as it floats on the iron in a heated chamber, and the purified slag is withdrawn. C. A. King.

Manufacture of pure iron. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 269,345, 10.5.26).—Iron formed by the decomposition of iron carbonyl, and containing carbon and oxygen, is melted or sintered in a high-frequency induction furnace *in vacuo*, and is afterwards exposed to an inert gas. C. A. King.

Protecting iron and steel articles against corrosion. S. G. S. Dicker. From Metals Protection Corp. (E.P. 269,305, 15.3.26).—As a protection against corrosion iron or steel articles are coated with a metal electropositive to iron, *e.g.*, cadmium or zinc, on which a second coating of a metal, *e.g.*, copper, electronegative to the first and preferably to chromium also, is applied. A final coating consists of chromium. The middle coating may be omitted, or the iron may be given an overcoating of chromium and an undercoating of a metal which is electronegative to both iron and chromium. C. A. King.

Apparatus for the production of metal [zinc] dust. R. B. Finn, Assr. to J. Finn Metal Works (U.S.P. 1,622,433—4, 29.3.27. Appl., [a], 28.4.26; [b], 12.6.26).—In an apparatus of the usual type for the production of zinc dust, the bottom of the condensing chamber is fitted with a scraping element to remove zinc oxide which first collects there. T. S. Wheeler.

Treatment of zinciferous materials. P. C. Rushen. From New Jersey Zinc Co. (E.P. 269,324, 10.4.26).—A briquetted charge of zinciferous material and a reducing agent is fed into an externally heated vertical retort of greater cross-sectional dimension than the length of the briquettes. The centre of the charge is heated mostly by convection of gases in the spaces between the briquettes. Zinc is volatilised and the retort is so worked that, without sintering, the briquettes disintegrate and may be drawn off at the bottom of the retort as a pulverulent residue. C. A. King.

Treatment of zinc-gold slimes. C. E. Meyer (U.S.P. 1,622,960, 29.3.27. Appl., 10.4.22. Conv., 16.4.21).—Zinc-gold slimes from the cyanide process for the recovery of gold are treated with potassium nitrate and steam, and the zinc oxide formed is extracted with ammonium carbonate solution and then recovered by removing ammonium carbonate from the solution with steam under pressure. T. S. Wheeler.

Nickel alloy. T. S. Fuller, Assr. to General Electric Co. (U.S.P. 1,623,948, 5.4.27. Appl., 16.2.25).—An alloy containing 60—75% Ni, 25—40% Cu, 0·5—5% Al, 0·4—0·03% C, 1—5% Mn, and about 2·5% Fe when forged below 1000° has a high tensile strength and elasticity. The higher aluminium content is associated with the lower carbon content and *vice versa*. T. S. Wheeler.

Resistor alloy. N. B. Pilling, Assr. to Westinghouse Electric & Manuf. Co. (U.S.P. 1,626,691, 3.5.27. Appl., 2.9.22).—An alloy containing 35—55 pts. of nickel, 65—45 pts. of copper, and up to 0·1% of magnesium is described, which has a negative coefficient of resistance. F. G. Crosse.

Treatment of metallic oxides. Aluminum Co. of America, Assees. of B. T. Horsfield (E.P. 248,360, 16.2.26. Conv., 2.3.25).—A stream of molten alumina, magnesia, or calcium oxide, free from silica, is blown with a gas at a velocity sufficiently high to form the oxide into hollow globules. In the preparation of alumina for electrolytic reduction to aluminium the natural raw material after electro-thermal reduction for the removal of associated oxide impurities contains an excess of carbon which is removed by disintegrating the molten stream by means of an air-blast. The granulated alumina is notably tougher than when solidified in the ordinary manner, and the product has superior properties as an abrasive. C. A. King.

Centrifugal amalgamator and concentrator. F. D. Lewis (U.S.P. 1,623,421, 5.4.27. Appl., 6.3.25).—A centrifuge for the treatment of gold-bearing gravels consists of three cylinders, which rotate about a common vertical axis. The gravel mixed with water is introduced into the innermost cylinder, and in passing through the apparatus to the external cylinder comes into contact with mercury maintained by centrifugal force in a number of annular grooves in the inner walls of the cylinders. The free gold particles amalgamate with the mercury. Means are also provided for separating and discharging through separate orifices light gravel and heavy concentrates containing mineral values other than free gold. T. S. Wheeler.

Amalgamator. N. S. Keith (U.S.P. 1,627,181, 3.5.27. Appl., 21.4.24).—A machine for effecting amalgamation consists of a rotating vertical shaft carrying a disc clamped to the shaft by a boss to which is fixed a vertical cylinder covered by, but insulated from, a lead casing. Concentric with this cylinder is a cup bolted to, but raised from, the disc. The cup contains a removable cylinder with an inner surface of copper and a serrated bottom. The whole is rotated, pulp containing mercury is supplied to the annular

space between the cylinders, and an electrical current is passed from the inner to the outer cylinder.
C. A. King.

Apparatus for washing refuse containing precious metal. G. Unrath (U.S.P. 1,627,129, 3.5.27. Appl., 18.5.26. Conv., 17.3.25).—Sweepings containing particles of precious metal are washed in a vessel of hemispherical shape having radial grooves on the inner side converging towards a central opening through which the heavier particles are discharged. A hollow stirring device agitates the muddy liquor in the cup, and lighter material is carried over into an outer container from which it flows into a settling tank. Clear water from the settling tank is lifted into the stirring arm for continuing the process with a fresh supply of refuse.
C. A. King.

Operating cupolas. F. K. Vial and G. S. Evans, Assrs. to Griffin Wheel Co. (U.S.P. 1,627,536, 3.5.27. Appl., 19.1.25).—Combustible gases formed inside a cupola are withdrawn and used to preheat air to be supplied to the cupola. C. A. King.

Separating magnetic from non-magnetic material. L. J. Ingolfsrud and W. H. Soule (U.S.P. 1,625,257, 19.4.27. Appl., 9.2.25. Conv., 31.3.24).—In a magnetic separator consisting of an endless belt which travels over a series of magnets, the belt is given a gradually increasing inclination lateral to the direction of motion. C. A. King.

Electrolytic refining of copper. Siemens & Halske A.-G. (E.P. 264,116, 6.3.26. Conv., 6.1.26).—In the electrolytic refining of impure copper, *e.g.*, copper bronzes and brasses, aqueous cuprous chloride is used as the electrolyte, and the liquor flowing from the bath is oxidised by means of air or chlorine to cupric chloride. The latter is reduced to the monovalent form again by bringing it into contact with a further quantity of the impure copper, thus maintaining the concentration of copper in the bath. After some time concentration of nickel, zinc, tin, or other impurity necessitates preferential electrolytic purification of the solution. Hydrochloric acid and colloids may be added to assist the process. C. A. King.

XI.—ELECTROTECHNICS.

High speed–high frequency inductive heating. E. F. Northrup (Trans. Amer. Electrochem. Soc., 1927, **51**, 497—513; cf. B., 1926, 283).—The construction of a high-frequency induction furnace suitable for melting 300 lb. of iron–nickel alloy in 20—25 min. is described with reference to illustrations and diagrams. Power is supplied by a 150 kw. generator at 900 volts and 1920 cycles, and the efficiency of the furnace is over 80%. For melting a charge of 250 lb. of nickel–iron alloy (casting at 1320°) the current consumption is about 53 kw.-hr. The crucibles of refractory sand are packed inside the copper induction coil with loose sand, which gradually builds up the crucible on the outside, whilst the melting operation wears away the inside; the average life of a crucible is 5—6 melts. Owing to the rapid rate of heating practically no impurities are introduced into the metal, and, in the case of alloys, efficient mixing of the constituents is obtained by the rotating action of the current on the molten metal.
A. R. Powell.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Protective coatings for duralumin and similar light-weight alloys for exposed construction. H. A. Gardner (U.S. Paint Manufrs. Assoc., Jan., 1927, Circ. No. 296, 1—26).—A comprehensive series of exposures (accelerated roof tests and sea-spray tests) on panels of duralumin, other light-weight alloys, and, for comparison, steel, covered with various types of protective coatings are fully described, and the results obtained over a period of 6 months are tabulated. The need for protection against the rapid form of surface- and intercrystalline corrosion to which the light-weight alloys are prone is stressed, and, owing to the greater degree of expansion and contraction of these metals compared with that of iron and steel, elastic linseed-oil paints are shown to be very suitable for the purpose. The use of basic pigments is, contrary to earlier opinions, highly desirable. Clear nitrocellulose or clear varnish coatings are not durable, but improvement is achieved by pigmentation and the use of a long-oil varnish vehicle. Aluminium powder has also given satisfactory results.
S. S. Woolf.

Painting galvanised iron conductor pipes: accelerated corrosion test. H. A. Gardner (U.S. Paint Manufrs. Assoc., Jan., 1927, Circ. No. 298, 51—64).—The causes of corrosion of galvanised iron rain pipes etc. are discussed. Pipes made from two grades of galvanised iron were painted with various described protective coatings, and semi-submerged at an angle in water, water containing resinous wood shingles of a corrosive nature, dilute sulphuric acid, and salt water, alongside untreated pipes in each case. The tabulated results of these accelerated corrosion tests indicate that painting the inside and outside surfaces of such pipes greatly increases their life. The salt-water test is regarded as too severe. S. S. Woolf.

Penetration tests on paste paints. E. F. Hickson (U.S. Paint Manufrs. Assoc., Jan., 1927, Circ. No. 300, 124—134).—The penetration test for asphalt (A.S.T.M. Standards, 1924, 944) as modified for lubricating greases (Proc. A.S.T.M., 1924, I, 518) is applied to paste paints ranging in consistency from stiff white lead-in-oil to soft runny pastes. Trials carried out with the 45° cone that replaced the needle in the original asphalt test, and with the later developed double-taper cone (90° cone with 30° point) show that a satisfactory classification into very stiff pastes, stiff pastes, medium stiff pastes, semi-pastes, and runny (thin) pastes is afforded.
S. S. Woolf.

Rapid method for the evaluation of Schweinfürth Green. D. Köszegi and S. Gerö (Chem.-Ztg., 1927, **51**, 303).—1 g. of the substance is dissolved in dilute sulphuric acid, the solution is made alkaline with potassium hydroxide solution, and heated, when the copper is reduced to cuprous oxide with the oxidation of the equivalent proportion of arsenite, which is determined by collecting and washing the cuprous oxide precipitate, dissolving it in ferric sulphate solution, and titrating

with potassium permanganate solution. The filtrate is neutralised with hydrochloric acid, excess of sodium bicarbonate added, and the arsenite remaining is determined by means of iodine and sodium thiosulphate. The total arsenic thus obtained is in good agreement with that determined by the distillation method. F. R. ENNOS.

Laboratory experiments on ethylene and propylene glycol ethers; their use in "odourless" brushing lacquers. H. A. GARDNER and A. W. VAN HEUCKEROTH (U.S. Paint Manufrs. Assoc., Feb., 1927, Circ. No. 302, 149—166).—A number of nitrocellulose lacquers, clear and pigmented, were made up, the nature and amount of resin, solvent, diluent, and plasticiser being varied in order mainly to examine the possibility of (*a*) replacing the strongly smelling butyl acetate type of solvent by ethers of propylene and ethylene glycols, (*b*) introducing rosin in place of more expensive resins, and (*c*) using light petroleum ("gasoline") as diluent in place of toluene, which has powerful solvent action on previously applied varnish etc. Satisfactory results were obtained on these lines, propylene glycol monoethyl ether being the best of the three ethers of this type investigated. The results of physical tests on the experimental lacquer films are tabulated and discussed. S. S. WOOLF.

Darkening of shellac solutions: effect of metals and solvents. H. A. GARDNER and S. LEVY (U.S. Paint Manufrs. Assoc., Mar., 1927, Circ. No. 304, 199—202).—Four types of shellac—clean dry bleached, superfine, fine orange, and T.N.—were each dissolved in five grades of denatured alcohol, and each solution was divided into four parts, to three of which were added bright iron, sheet tin, and granulated zinc, respectively, the fourth being used as a blank. The four sets of tests, in glass jars with paper-insulated caps, were shaken daily for 5 min. over a period of 6 months. The presence of metal was shown to cause a gradual darkening, tin being worse than iron, and zinc being the least harmful in this respect. The grade of alcohol seemed to affect the colour to some extent, particularly that containing pyridine, but the darkening effect was less marked than that due to metal. An increase of viscosity was also noted in the solutions containing tin, and to a lesser but still considerable extent in those containing iron and zinc. It is advisable that shellac be "cut" in apparatus containing little, if any, metal, and be stored in glass containers. S. S. WOOLF.

Influence of driers on the properties of some ester gum and synthetic resin varnishes. G. G. SWARD and H. A. GARDNER (U.S. Paint Manufrs. Assoc., Feb., 1927, Circ. No. 301, 135—148).—Various amounts and combinations of lead, manganese, and cobalt linoleates were added to varnish bases prepared by cooking equal pts. by wt. of oil (3 linseed oil : 1 tung oil) and resin at 264°, until requisite body was reached. The time required for this was 9 hrs. when ester gum was the resin used, and 3½ hrs. in the case of a synthetic resin derived from phenol, formaldehyde, and colophony. The varnishes were thinned to 50% non-volatile content, and tested for drying time, viscosity, colour, elasticity, hardness, resistance to solvents, keeping qualities, durability, and behaviour when made up into enamels. The tabulated results indicate the influence exerted by kind and concentration of drier on the type of varnish under consideration. Large amounts of driers tend to produce brittleness, and do not accelerate drying. Manganese driers reduce the elasticity of varnish films, and induce skinning on storage of the varnish to a greater extent than do lead or cobalt driers, which are preferred for enamels. The advantages accruing from entire omission of driers might be obtained in varnishes intended for any exterior uses where slow attaining of final hardness is not a drawback. S. S. WOOLF.

Pigment finishes. VOGEL.—See XV.

PATENTS.

Converting into soluble form insoluble condensation products of carbamide or a derivative thereof and formaldehyde. SOC. CHEM. IND. IN BASLE (E.P. 246,127, 4.1.26. Conv., 17.1.25).—Hard condensation products of carbamide or its derivatives and formaldehyde, particularly waste splinters or shavings from the working of these materials, are converted into soluble form by heating them under pressure at above 100° with excess of formaldehyde. The latter is distilled off, or further condensed with a fresh quantity of carbamide etc. S. S. WOOLF.

Preparation of alcohol-soluble, bromine-containing condensation products. A. JÜDEFEIND (G.P. 436,819, 9.10.23).—Tannin, brominated before or after the condensation, is condensed with carbamide or its derivatives and formaldehyde in the presence of a condensing agent, the product being used for dermatological purposes. S. S. WOOLF.

Production of phenol-aldehyde resins. BAKELITE G.M.B.H. (E.P. 246,834, 22.1.26. Conv., 27.1.25).—Phenol–aldehyde condensation products are heated with suitable quantities of alkali in aqueous solution (amounts to be determined by preliminary experiments). They are then "salted out" in gel form with sodium chloride, alkali carbonates, magnesium sulphate, alums, etc. In addition, hydrotropic salts, preferably so selected as to form soluble or insoluble compounds with the salts used for precipitating the resins, may be present. Typical hydrotropic salts are sodium salicylate, alkali metal soaps, or salts of fatty acids, of sulphonated-, chlorinated-, or hydroxy-fatty acids, or of resin acids. After washing and drying, the resins salted out in this way are powders having m.p. higher by 100° or more than the original phenol–aldehyde resins. S. S. WOOLF.

Hardening phenol–aldehyde condensation products under heat and pressure. G. M., and N. G. HICK (E.P. 269,037, 12.8.26).—A sealed pressure-tight vessel containing phenol–aldehyde condensation products (moulded in plaster of Paris) and a volatile liquid, *e.g.*, alcohol, is immersed in a boiling liquid, *e.g.*, a saturated solution of sodium chloride, the temperature and pressure inside the vessel being thus determined. Gas under pressure may be substituted for the volatile liquid. Different types of pressure vessels are detailed. S. S. WOOLF.

XV.—LEATHER; GLUE.

Tanning, dyeing, and finishing formaldehyde-tanned leathers and skins. J. W. Lamb (Halle aux Cuirs, 1926, 6—16; Chem. Zentr., 1926, II, 2650—2651).—Every 15 min., 7·2 kg. of sodium carbonate and 4·5 litres of 40% formaldehyde in 36 litres are added in 4 portions for every 100 kg. of pelt in 225—270 litres of water at 35°. The tannage is complete in 3—6 hrs. with light skins, and in ½—2 days with heavy hides. The temperature may be increased to 50° towards the end of the tannage. The leather is then treated with a 1% ammonium sulphate solution, horsed up, and then drummed with a solution of 2·25 kg. of soft or neutral soap and 2·25 kg. of sodium chloride at 50° for 2—3 hrs., giving a longer time for heavy leather. An emulsion of fish or olive oil with soap can also be used, and then the salt solution must be given afterwards. The addition of alkali during the tannage prolongs the process; an excess of formaldehyde causes brittleness. For dyeing with basic dyes, the leather is mordanted with 6% chrome alum and 0·75% sodium carbonate (on dry weight) and dyed with addition of sodium carbonate and 5—10% of sodium chloride or sulphate. Chrome leather dyes (direct) can be used if sodium bisulphate is added to the dye liquor. Acid dyes can be applied in the drum or with a brush if soap and formaldehyde are mixed with the dye solution. For deeper shades sodium chloride is added. The fastness to washing can be increased by dipping the dyed leather in a solution of potassium dichromate, copper sulphate, or acetic acid for a short time. Unfixed dye is removed by washing and soap. Previous treatment with ammonium acetate or formate is recommended when dyeing with acid dyes.

D. Woodroffe.

Pigment finishes. W. Vogel (Collegium, 1926, 560—562).—Pigment finishes consist of three ingredients chiefly, viz., pigment, binding agent or carrier, and the solvent. Both inorganic pigments and organic pigments (colour lakes) are used. Fats, proteins, and carbohydrates are used as carriers. Linseed oil and varnishes are used as carriers in patent leather manufacture. Aqueous solutions of glue, egg albumin, and casein are used for this purpose; also aqueous solutions of gums, dextrin, and starch. There are two kinds of pigment finishes used in leather finishing, viz., albumin finishes containing egg albumin in aqueous solution and the collodion finishes, with nitrocellulose as the binding agent in organic solvents. The albumin finishes cover the leather well and give an even colour without detriment to the appearance or feel of the leather. The waterproofness leaves much to be desired, and where it is essential, the collodion finishes must be used. Collodion finishes are made by dissolving collodion wool in organic solvents, grinding with pigments usually together with a small amount of castor or olive oil to make it elastic. Both types of finish can be used for making splits into imitation grain leathers. The practical value of these finishes can be tested by applying them to leather, drying out, and testing for fastness to light, rubbing, and water, also the feel. The fastness to light is exceptionally good in most of the commercial finishes. Collodion-finished leathers are satisfactory to rubbing and to water. They have not such a smooth feel as leathers finished with the albumin finishes.

D. Woodroffe.

XVII.—SUGARS; STARCHES; GUMS.

Luminescence of sugars and of sugar factory products. K. Šandera (Z. Zuckerind. Czechoslov., 1927, **51**, 237—245).—Luminescence in sugar factory products is due, not to the ash nor to the colouring matter, but to a colourless or but slightly coloured substance, soluble in ether and chloroform, one of the first products of the caramelisation of sugar or of the destruction of invert sugar. Beet products exhibit it to a greater extent than cane products. An apparatus is described for its measurement. J. P. Ogilvie.

Determination of hydrogen-ion concentration of sugar factory products. O. Spengler and F. Tödt (Z. Ver. deut. Zuckerind., 1927, 115—118).—Wulff's method (Chem.-Ztg., 1926, **50**, 732) applied to a beet molasses at 30° and a liquor at 50° Brix required 5 and 10 min., respectively, before the final indication could be obtained, and is considered suitable only for liquids that are not more viscous than water. For routine practice in the sugar factory the colorimetric drop method is preferred. J. P. Ogilvie.

Determination of amides in sugar factory products. J. Vondrák (Z. Zuckerind. Czechoslov., 1927, **51**, 261—271).—As Schulze's method gives results lower than the truth in the presence of sugar, a process is recommended depending on the principle that amides, ammonium salts, and most of the amino-acids are entrained by the precipitate formed on the addition of sodium carbonate to mercuric acetate. In determining the original ammonia and the amides, respectively, the mercuric precipitate is distilled in the presence of magnesia, magnesium sulphate, and sodium thiosulphate before and after hydrolysis by acid. J. P. Ogilvie.

Decolorising carbon [in sugar] work. S. Kühn (Z. Zuckerind. Czechoslov., 1927, **51**, 271—272).—A consideration of the question whether it is advantageous to filter the liquors previous to their treatment with decolorising carbon, leads to the following figures for the insoluble matter in liquor taken directly from the blow-ups being given: organic matter, 3·77, and inorganic, 7·12 kg. per 1000 quintals of sugar introduced in 24 hrs. J. P. Ogilvie.

Decolorising carbons and their action on molasses. M. Garino and G. Benvenuto (Giorn. Chim. Ind. Appl., 1927, **9**, 169—175).—Of a number of samples of commercial decolorising carbons, carboraffin gives the best results for decolorising either molasses solutions or methylene-blue solutions. Molasses contains different colouring matters, each of which exhibits a definite degree of affinity for any particular adsorbent carbon. Adsorption of the various colouring substances obtainable from caramel differs greatly from that of the substances formed by the action of lime on invert sugar, these being, according to Peligot, mainly glucic and *apo*glucic acids. T. H. Pope.

Invertase present in [beet] sugar factory waste-waters. C. L. Matoušek (Z. Zuckerind. Czechoslov.,

1927, **51**, 213—220).—Invertase resulting from the micro-organisms introduced by insufficiently washing the roots may, when the water used in the diffusion battery is impure, and especially in the case of returned waste waters, give rise to a relatively considerable invert sugar formation. At a temperature of 36° the invertase present in a very dirty water, formed, in a 5% sucrose solution, invert sugar to the amount of 1·36—1·95% in 24 hrs. J. P. OGILVIE.

Uniformity in sugar beet analysis. A. W. LING (Int. Sugar J., 1927, **29**, 86—88).—Recommendations as to sampling are made. Dirt is determined by the usual method of washing and re-weighing, and in order to obtain the sample of pulp for the sugar determination the whole of the 25 roots should be taken. A circular saw revolving at 1000 r.p.m. is used to obtain the pulp, this being driven so that it rotates away from the beet, the resulting fine pulp being collected in a hopper beneath the saw, and well mixed. In determining the sucrose content, the Krüger method may be used, but if a less efficient method of pulping has been employed (viz., a mincing machine) it is then necessary to apply a hot water digestion process, *e.g.*, the Pellet. J. P. OGILVIE.

Content of sulphur dioxide in German consumption sugars. O. SPENGLER and C. BRENDEL (Z. Ver. deut. Zuckerind., 1927, 167—173).—Direct titration with 0·005*N*-iodine solution affords a useful sorting-out test, the end-point generally, though not always, being sharp. Useful results were obtained by the sulphide-stain method (cf. B., 1927, 312), slightly modified. Of 39 samples of white consumption sugars examined only 5 gave over 10 pts. per million of sulphur dioxide, the highest being 30 p.p.m. Ultramarine when present does not affect the iodine titration. A sample of sugar free from sulphur dioxide on being strongly blued with ultramarine, showed 17 p.p.m. by the sulphide-stain method. J. P. OGILVIE.

XVIII.—FERMENTATION INDUSTRIES.

Detection of fruit wine in grape wine. A. HEIDUSHKA and C. PYRIKI (Chem.-Ztg., 1927, **51**, 442).—Röttgen's reaction for the detection of fruit wine in white wine is uncertain if any quantity of blue grapes has been used in its manufacture or if the wine is adulterated with decolorised red wine. A negative result in the test, however, indicates the certain absence of fruit wine, but there is still no sufficiently sharp reaction for the certain detection of the latter in white wines (cf. B., 1927, 264). A. R. POWELL.

Spectrometric detection of fruit wine in wine. O. E. KALBERER (Z. Unters. Lebensm., 1927, **53**, 208—221).—The absorption spectra of various red and white wines, with and without added fruit wine, have been measured photographically and represented graphically by plotting the thickness of the layer against the number of waves per mm. There are characteristic differences between grape and fruit wines. The former show an increase in general absorption with decrease of wave-length, with distinctive absorption bands between 34,000 and 38,000, the greatest absorption occurring at about 36,800—38,100. Some grape wines also show an absorption band between 29,000 and 31,000. With fruit wines, the latter band is strongly marked at 27,000, and these wines are further distinguished by a band in the region 40,800—43,000, which is scarcely noticeable with grape wines. A formula has been deduced from which the extinction coefficients of wines can be calculated, and the curves obtained by plotting logarithms of these coefficients against reciprocal of wave-lengths agree closely with those determined experimentally. It is shown that by comparison of the extinction curve of a mixed wine with that of a pure wine, it is possible to detect and determine the amount of any addition of fruit wine. H. J. DOWDEN.

Determination of degree of acidity of wines with diazoacetic ester. G. BREDIG and K. SIEBENMANN (J. pr. Chem., 1927, [ii], **116**, 118—128).—Preliminary experiments were carried out on the hydrolysis of diazoacetic ester (cf. Bredig and Fraenkel, A., 1905, ii, 692; Fraenkel, *ibid.*, 1907, ii, 746) with 0·0021*N*-nitric acid and 0·0025*M*-tartaric acid, at 25°, in aqueous-alcoholic solution (alcohol content, 3—18% by vol.). The values for the ratio of the velocity constant (k), and hydrogen-ion concentration in mol./litre (h) are tabulated, and they are found to decrease in magnitude with increasing amount of alcohol. The inversion of sugar, catalysed by 0·002*N*-hydrochloric acid and 0·0025*M*-tartaric acid, at 75·86°, in various aqueous-alcoholic solutions has also been studied, values for k and h being determined. The percentage decrease of hydrogen-ion concentration ($dh/h \times 100$) of dilute solutions of malonic, tartaric, and acetic acids, on adding alcohol, has been determined by the following methods: (*a*) electrometrically at 18°, (*b*) diazoacetic ester, (*c*) sugar inversion. The results from methods (*a*) and (*b*) are in good agreement; those from (*c*) are distinctly higher. The degree of acidity (no. of mg. of hydrogen ion per litre $= h \times 10^3$) has been determined for four wines—white Gaillard (1913 and 1922), white Burgundy (1921), and Portuguese red (1923)—by methods (*a*), (*b*), and (*c*). In method (*a*) a hydrogen|wine|saturated calomel cell was used. The results from (*a*) and (*b*) are in close agreement, whilst those from (*c*) are slightly lower. H. BURTON.

Determination of methyl alcohol in alcohol and alcoholic beverages using the immersion refractometer. J. F. WILLIAMS (Ind. Eng. Chem., 1927, **19**, 844—845; cf. Leach and Lythgoe, A., 1905, ii, 655).—The distilled sample is accurately diluted to 20% of total alcohol by volume, and determinations made of the sp. gr. and refractive index, the % of total alcohol indicated by the sp. gr. of the diluted solution being slightly greater than 20% if methyl alcohol is present, since the densities of the two alcohols on dilution do not run strictly parallel. From the difference between the refractometer scale reading and the % of total alcohol the amount of methyl alcohol is obtained by reference to a table. The results are accurate to within 0·5% with solutions containing 1% or more of methyl alcohol, but smaller quantities are best determined by Wright's colorimetric method (cf. A., 1927, 687). W. J. POWELL.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPTEMBER 16, 1927.

I.—GENERAL; PLANT; MACHINERY.

Priming of saline waters [in locomotive boilers]. A. F. JOSEPH and J. S. HANCOCK (J.S.C.I., 1927, **46**, 315—321 T).—Quantitative determinations of the percentage of priming were made with an experimental boiler working at 150 lb. pressure, the steam with priming water being trapped and condensed and the total dissolved solids determined. The amount of priming was found to increase with the rate of evaporation, but to decrease with increase of pressure. Sodium sulphate, sodium carbonate, sodium chloride, and calcium chloride in solution were found to promote priming in the order mentioned. No evidence was found of any effect from calcium carbonate or boiler scale in suspension. The water level in the boiler is important. The known action of castor oil in preventing priming was confirmed, a 1 in 6 emulsion in 1% soap solution being used.

C. IRWIN.

Rate of growth of crystals in aqueous solution. G. H. MONTILLON and W. L. BADGER (Ind. Eng. Chem., 1927, **19**, 809—816).—The rate of growth of crystals of sodium sulphate and magnesium sulphate during continuous crystallisation was studied. The hot solution, to which a given weight of sized seed crystals was added at regular intervals, was passed, at a temperature at which crystallisation was just commencing, into a long horizontal tube with stirrer. The crystals made during a run were dried, weighed, and screened. The results showed that growing small crystals do not all increase in size at the same rate, but those somewhat larger increase in weight faster than the smaller ones. Under given conditions there is a definite relation between increase in weight and increase in surface. The greater the time of contact in the crystalliser, the larger were the crystals, an arithmetical relationship being observed. The variation in the rate of growth with temperature (between 27° and 31°) and viscosity was also studied. From the data obtained it is possible to predict the proportion of crystal sizes for given conditions of crystallisation. C. IRWIN.

Protecting the eyes of chemical workers. J. E. HANNUM (Ind. Eng. Chem., 1927, **19**, 817—819).—Features of goggles most suitable for workers of various types of chemical processes are described. A list of a large number of substances injurious to the eyes, with the symptoms produced, is given. Methyl alcohol and lead are considered particularly dangerous, the latter causing atrophy of the optic nerve. C. IRWIN.

Conditions of flow into the vertical capillary tube of the Saybolt thermo-viscosimeter. W. H. HERSCHEL (Ind. Eng. Chem., 1927, **19**, 837—840).—In the Saybolt viscosimeter the time required to fill a vertical capillary tube immersed in the liquid between two given marks is measured. $d^2pgt/32 = [(\mu - \mu_a)(h + c) + \mu_a L] \log_e(b + c)/(a + c) - (b - a)(\mu - \mu_a) = \gamma$, where t = time of flow, μ and μ_a are the viscosities of the liquid and of air, h = height of liquid above the bottom of the capillary in the jar, b and a are the depths of the meniscus below the surface at the beginning and end of the run, d = diameter and L = total length of capillary, and c = capillary rise = $4\gamma/pgt$, where γ = surface tension. A large number of tests were made with two instruments and compared with viscosity tests by the Bingham viscosimeter. To obtain good agreement it was found necessary to use the figure 29 in place of 32 in the above equation to compensate for the error due to drainage effect from the tube. C. IRWIN.

Technical aerosols and their characteristics. P. BEYERSDORFER (Kolloid-Z., 1927, **42**, 229—233).—A treatment of the technological problems connected with the formation, or prevention of formation, of the air dispersoids dust, smoke, and fog. Examples are given to illustrate the influence of the composition of both the solid and the gaseous phase on the properties of the aerosol. The question of dust explosions is considered in some detail, with special reference to the explosion of mixtures of sulphur and sugar dust, and to coal dust explosions and methods available for their prevention. The removal of noxious fumes and harmful gaseous suspensions is regarded as a problem of sol–gel transformation. A few examples of aerosols in which one phase is fluid are discussed. L. L. BIRCUMSHAW.

Silicic acid gel and its adsorbability. P. MAUTNER (Kolloid-Z., 1927, **42**, 273—275).—The manufacture of silicic acid gel from water-glass is briefly described, and the effect of heating the gel is discussed. Tables are given to show the dependence of the adsorptive power of the gel on the drying temperature, and thus on the water content. From figures showing the percentage of aniline, phenol, and acetic acid adsorbed from various solvents it appears that the adsorption is least when the solvent is water. The various technical uses of the gel as catalyst, catalyst-carrier, adsorber of vapours, refrigerating agent, etc. are reviewed, and a description is given of the method used by the Silica Gel Corporation for the recovery of benzene from coke ovens.

L. L. BIRCUMSHAW.

Adhesives and adhesion: relation of joint strength to tensile strength of films. J. W. MCBAIN and W. B. LEE (J.S.C.I., 1927, **46**, 321—324 T).—The tensile strength of joints between ferrotype or celluloid of different grades of sodium silicate was determined.

The results are affected by the atmospheric humidity but the relative order of strengths was independent of the class of material joined so long as it was smooth. Sodium silicate joints are very brittle, and the quality of brittleness or "deformability" is very important. The tensile strength of the adhesive is no guide to the strength of joint between smooth surfaces. The strength of fibres of gelatin glue diluted with dextrose or glycerin between plates was determined. Dilution weakens the films but increases deformability. "Glue pearls" were found to be inferior to ordinary glue in both respects. Shear tests on walnut joints with diluted glues gave results similar to the strengths of the adhesive, and this was also true for tension tests. Minor variations were found with the direction of the grain and the rate of loading. The mechanical explanation of the adhesion of glued wood joints (*i.e.*, joints between rough surfaces) is supported by the similarity of joint strength with sodium silicate and glue diluted to the same tensile strength. C. Irwin.

Mathematical methods of frequency analysis of size of particles. R. P. Loveland and A. P. H. Trivelli (J. Franklin Inst., 1927, **204**, 193—217).

Collapsing strength of thin tubes and pipes in chemical works. W. F. Schaphorst (Ind. Eng. Chem., 1927, **19**, 887).

Pipe still. Ashworth.—See II.

Recorder for carbon dioxide. Rosencrans.—See XVI.

Patents.

Furnaces for heating. British Furnaces, Ltd., and E. W. Smith. From Surface Combustion Co. (E.P. 274,003, 23.3.27).—The furnace comprises a trough of refractory material through which the goods to be heated (*e.g.*, steel for cementation in boxes) are pushed. The length of the furnace is divided into two parts. In the cooler or feed section fuel is burnt in passages under the trough, and the products of combustion pass up between the sides of the trough and the outer wall, and then over the goods in contact with them to an outlet or chimney at the feed end. In the other or hotter section fuel is burnt in passages under the trough as before, but the products of combustion pass upwards between the sides of the trough and the outer wall, and are deflected towards the outlet end of the furnace, and then pass the full length of the furnace between an outer and inner roof, the latter forming a muffle, so that when the goods have attained the required temperature they are in a still, inert atmosphere. B. M. Venables.

Heat exchanger. J. W. Young, and Metropolitan-Vickers Electrical Co., Ltd. (E.P. 273,886, 16. and 24.6.26).—The apparatus comprises a number of annular spaces, usually horizontal, through half of which the hot fluid, which may be furnace gases after leaving economisers, passes from an external supply trunk round nearly the whole circumference of the spaces to a common, axial outlet flue, which may conveniently be a chimney or the continuation of one. The fluid to be heated (air) passes through the alternate spaces in the other direction, both supply and delivery trunks being external to the cylindrical casing. In the case of dusty gases the whirling motion causes the dust to pass to the outer circumference, and it may be caught by slits with inturned lips situated just before the outlet, but in the outer circumference. B. M. Venables.

Method of transforming heat applicable to refrigeration. R. W. Davenport (E.P. 258,847, 12.8.26. Conv., 25.9.25).—The process operates on the facts that an inert gas expanded or bubbled briskly through a volatile liquid will cause evaporation, and that the gas–vapour mixture on compression will deposit liquid. A suitable form of the apparatus is described in which a supply of a substance which is liquid at ordinary atmospheric temperature and pressure, but volatile, circulates in an expansion or refrigerating vessel, heat-exchanging conduits, and gas-lift circulating pump. An insoluble gas such as air is expanded in the first vessel, and passes to the pump, where it is compressed and the liquid produced transferred to the main body of liquid while in the air lift, the last-mentioned being designed to operate gently so as to avoid evaporation as much as possible. B. M. Venables.

Adsorption apparatus. Silica Gel Corp., Assees. of F. B. Krull (E.P. 255,819, 29.3.26. Conv., 27.7.25).—The absorber is divided into a number of zones, each of which is put into communication with the inlet and outlet for either fluid to be treated or regenerating fluid, each in turn, by means of one or more continuously revolving plate valves. B. M. Venables.

Carrying out exothermic chemical reactions under pressure and at a high temperature. L'Air Liquide Soc. Anon., Assees. of Soc. Chim. de la Grande Paroisse (Azote et Prod. Chim.) (E.P. 268,721, 25.10.26. Conv., 31.3.26).—The heat from exothermic reactions is prevented from damaging the outer pressure-resisting vessel by passing a current of gas between the outer and catalyst vessels. The gas may be the gases before reaction, or after reaction if they are taken outside and cooled before returning, or a separate gas may be used. B. M. Venables.

Apparatus for evaporating liquids. W. Wiegand (E.P. 272,703, 23.7.26).—An apparatus for evaporating liquids of high viscosity and containing solid matter, such as fruit syrups, comprises a sloping bank of heating tubes and a separate evaporating chamber; a centrifugal pump circulates the liquid from the lower end of the evaporating chamber, through the tubes up the slope, and through an adjustable door to the other end of the evaporating chamber. The adjustable door or valve may be spring-controlled, and is intended to maintain pressure in the heating tubes so that no evaporation takes place there, hence there is less risk of formation of scale. B. M. Venables.

Method of working steam-distillation plants. Metallbank & Metallurgische Ges. A.-G., and W. Gensecke (E.P. 273,092, 23.6.26).—A portion of the liquid is distilled, and the whole is lifted, by bubbles of a gas (*e.g.*, part of the "driving-off" steam), and the liquid thus lifted falls down a series of trays in contact with a con- or counter-current of "driving-off" steam. The driving-off steam may be used several times in

separate vessels, but for the lifts live steam can be used only once. B. M. Venables.

Absorption refrigerating or heating machine. Siemens-Schuckertwerke G.m.b.H. (E.P. [a] 250,593, 7.4.26. Conv., 8.4.25, and [b] 259,985, 15.10.26. Conv., 17.10.25).—(a) The gas evolved in either the boiling-off or the evaporation side of a refrigerating system is caused to circulate the liquid in the other side, by means of bubbles in an ascending pipe. (b) From the boiler a mixture of liquid solvent and gas ascends to a gas-separating chamber, and the gas is finally freed from any vapour of the solvent by heat interchange with the circulating solvent in a vessel at a lower level than the gas-separating chamber. B. M. Venables.

[Cooling attachment for] rotary kilns. J. S. Fasting (E.P. 272,616, 18.3.26).—The clinker or material that has been heated leaves the kiln through ports in the circumference and passes to a number of individual tubular coolers arranged round the kiln and extending beyond the lower end. The further end of every tube is partially closed by an inturned flange, preventing exit of material but allowing entry of cooling air, which passes through the tube into the kiln and is used for combustion. The outlet for material from each tube is in the form of a tangential shoot which delivers the material at about the same height as the axis of the kiln. To aid the transmission of heat from the material to the air, loose chains may be provided which are alternately buried and exposed to the air. Large pieces of clinker which will not enter the coolers are discharged from time to time through swinging doors closing the end of the kiln and surrounding the fuel inlet. B. M. Venables.

[Hydraulically] grading solid substances contained in liquids. Soc. Anon. des Manuf. des Glaces et Prod. Chim. de St.-Gobain, Chauny, et Cirey (E.P. 252,683, 10.4.26. Conv., 28.5.25).—An upward-current classifier wherein the pulp and classifying water are fed together (as thin pulp) into the lower conical or pyramidal part of the apparatus and pass upwards into a portion of uniform cross-section, where the flow is maintained uniform over the whole horizontal cross-section by means of honeycomb or tubular baffles. The oversize is withdrawn from a bottom spigot, and the undersize and the bulk of the water overflow at the top into a regulating weir with a waste flow to maintain a constant level of feed to a similar vessel, where a finer product can be collected, and so on through as many vessels as desired. B. M. Venables.

Mixing machine. M. Nairn (E.P. 273,101, 5.7.26).—The machine is constructed with a series of hook knives running in close proximity to a feed roller which protrudes into the feed passage or hopper on the side to which the material is dragged by the knives. The roller is rotated in the opposite direction to the knives either by gearing or by the drag of the material, and may be made hollow for heating purposes. The roller is mainly to increase the rate of feed; the main part of the mixing action is effected against the stationary casing as usual. B. M. Venables.

Grinding or disintegrating machines. H. J. Denham, and H. Simon, Ltd. (E.P. 473,499, 12.7.26).—Two shafts are rotated in the same direction at exactly the same speed. The beaters on each shaft are alternately long and short, the long beaters on one shaft coming opposite the short beaters on the other. B. M. Venables.

Apparatus for grinding or comminuting rubber or other materials. C. E. Gardner (E.P. 272,572, 19.2.26).—A slab of rubber, wood, bark, or similar material is fed between positively driven feed rollers (at least two above and two below) against the surface of an abrading drum. The latter is perforated between the ridges or teeth, so that air can be drawn through to keep the material cool and remove the powder, and a rotary brush runs against the further side of the abrading drum to keep the perforations clear. At starting or stopping the positive drive may be taken off the feed rollers, so that they may be worked by hand. B. M. Venables.

Centrifugal impact pulveriser. F. E. Agnew, Assr. to M. H. Brotherton (U.S.P. 1,635,453, 12.7.27. Appl., 9.12.25).—The material passes in succession through a funnel-shaped section of the outer casing, on to a horizontally-rotating disc with upstanding radial blades, from which it is flung to a cylindrical impact section of the outer casing, then through another funnel-shaped section, and so on downwards over every disc. B. M. Venables.

Atomising liquid and semi-liquid materials. F. Wreesmann (E.P. 269,774, 21.8.26).—The liquid is sprayed by a centrifugal rotor, the outlets from which are in the form of slots with their long side parallel to the axis, the size of the slots being adjustable. Air or gas is supplied under pressure (produced by the same machine, if desired) through, below, or above the rotor, any or all. B. M. Venables.

Separation and recovery of gases and vapours by solid absorbents. A. Godel (E.P. 267,369, 10.6.26).—The absorbent is contained on a porous plate, and within a sheet metal lining, which provides an annular space between the absorbent and the external wall of the absorber. Gas to be treated is passed through the absorbent until it is saturated with the constituent it is desired to recover. To regenerate the absorbent the absorber is isolated and heated by steam coils or electrical elements disposed between the absorbent container and the external wall of the absorber. During the heating the permanent gas in the absorber is kept in forced circulation by a steam injector situated at the base of the layer of absorbent. The injected steam forms only a small proportion by volume of the gas in circulation, consequently no excessive condensation of water takes place during the heating stage. Heat is efficiently transferred from the heating elements to the absorbent by convection. S. Pexton.

Centrifugal apparatus for dust separation. J. Keith & Blackman Co., Ltd., and G. Keith (E.P. 273,475, 18.6.26 and 9.3.27).—The gas is caused to form a vortex in a cylindrical casing by admitting it tangentially to the circumference but obliquely to the axis. An enlarged separating zone is provided remote (axially) from the inlet end of the casing. The outlet for clean gas is at the axis in the enlarged zone, and is either through a flared end of, or through longitudinal

slits or louvres in the wall of a pipe, so as to remove clean gas from the interior of the vortex over a considerable axial length. In each case internal vanes are provided to destroy the rotational movement of the gas without undue shock. B. M. VENABLES.

Indicating the exhaustion of substances used for cleaning gases. DEUTS. GASGLÜHLICHT AUER-GES.M.B.H. (E.P. [A] 272,855, [B] 272,858, 1.3.27. Conv., 17.6.26).—(A) An easily recognisable substance is added to the gas which will be removed by the cleaning filter in a similar manner to the noxious impurities that must not be allowed to pass; *e.g.*, air containing unrecognisable but dangerous nitrous gases may have an addition of easily smelt acetic acid. (B) A substance is added to the filter itself (or just before) of such a nature that on combining with the first traces of noxious substance passing the filter a third easily recognisable substance will be formed; *e.g.*, moisture which will inhibit the action of some filters may be recognised by admixture of calcium carbide, or nicotine may be used to indicate the presence of hydrocyanic acid. B. M. VENABLES.

Apparatus for hydrogenation of liquids. G. R. SCHUELER (E.P. 273,045, 31.12.26).—The apparatus comprises a number of cages containing the catalyst assembled on one or more perforated tubes through which the hydrogen is supplied. B. M. VENABLES.

Polarimeters. E. LEITZ (E.P. 271,824, 9.3.27. Conv., 31.5.26).—A polarimeter with half-shade device is provided with one or more glass plates of which the inclination can be varied to neutralise the rotation produced by the substance to be measured. The plane of incidence on the glass plates should make an angle of 45° or 135° with the bisecting line of the half-shade device. A quartz wedge may also be incorporated. B. M. VENABLES.

[Multiple-tier gas] ovens for baking, drying, and other heating operations. T. & T. VICARS, LTD., and E. M. CROSLAND (E.P. 273,861, 4.1.26).

Observation window for furnaces. VASTINE ENGINEERING PRODUCTS CORP. (E.P. 257,266, 17.8.26. Conv., 24.8.25).

Absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKT. (E.P. 255,119, 13.7.26. Conv., 13.7.25).

Method and apparatus for refrigeration. SILICA GEL CORP., Assees. of E. B. MILLER and W. L. EDEL (E.P. 249,109, 4.3.26. Conv., 11.3.25).

Extinguishing fires by foam. W. GRAAFF (E.P. 274,574, 24.4.26).

Screens of the rotary type for separating various sizes of granular material such as ore, coal, etc. M. C. DUNSMORE and D. W. RITCHIE (E.P. 274,270, 6.7.26).

Separation of condensate from steam. R. F. CHEVALIER (E.P. 274,531, 20.2.26).

Wet separating apparatus. J. SPROUL (E.P. 274,595, 8.2.27).

Drying apparatus (E.P. 267,784).—See II.

Filtering apparatus (E.P. 273,364).—See II.

Continuous distillation (G.P. 439,712).—See II.

Fractional distillation (Austr. P. 104,137).—See III.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Colloid chemistry of coal and related problems. H. WINTER (Kolloid-Z., 1927, **42**, 233—242).—An account is given of evidence obtained by numerous investigators for the colloidal nature of coal, with special reference to previous microscopical studies of the author (cf. *ibid.*, 1916, 8; B., 1925, 194, 605), and to the work of Duparque (cf. A., 1926, 380). The relation of dopplerite to mineral coal is fully discussed. L. L. BIRCUMSHAW.

Displacement of moisture in lignite by oils. E. BERL and A. IMMEL (Koll. Chem. Beih., 1927, **24**, 181—267).—The hydrophilic properties of fuels depend on the degree of carbonisation undergone during their formation. Mineral coal (in which most of the hydroxyl groups present in the original cellulose have disappeared) has a pronounced hydrophobic character, whilst peat has a high capacity for combining with water. Lignite occupies an intermediate position in this respect. Attempts to obtain a fuel of high value by removing water in the liquid form from raw lignite are reviewed. Experiments were carried out on the displacement of the water by oil. The raw lignite mixed with an oil immiscible with water was subjected to a pressure of about 150 kg./cm.^{2} At room temperature it was possible to remove about 27—30% of the bound water in one operation, but 41% could be removed by working at 75°. By repetition of the process a large number of times the water content can be reduced to 6%. The presence of alkali causes the lignite to become more hydrophilic, owing to the formation of humates, and thus affects the displacement adversely. Conversely, the presence of acid favours the displacement of water by oil. Neutral salts have little effect. Similar effects were observed in flotation experiments, using benzene and water as the pair of liquids. The displacing capacity of the light oil used may be increased by addition of a liquid slightly miscible with water, such as aniline. No relation could be found between the displacing power of the oil and its surface tension or viscosity. The basic character of aniline and its homologues is not the cause of the good displacing power, for a typical acidic oil, *m*-cresol, is an excellent displacing agent. It appears that in the best displacing agents a hydrophilic radical (amino, hydroxyl, etc.) is combined with a hydrophobic residue. The facts are discussed in the light of the theories of Hardy, Langmuir, and Harkins. E. S. HEDGES.

Microstructure of typical New Zealand lignites. W. P. EVANS (Fuel, 1927, **6**, 368—373).—The 28 photomicrographs of etchings and sections of lignites show the predominance of coniferous wood and the comparatively rare occurrence of spores. There is evidence that some of the coal-forming trees were of araucarian type, whilst other etchings show structures similar to those of the kauri pine. The influence of igneous intru-

sions on the structure is illustrated, the coal in one case being half coked and full of pores.

H. D. Greenwood.

Decomposition of vegetable matter under soils containing calcium and sodium as replaceable bases. E. M. Taylor (Fuel, 1927, **6**, 359—367).—The possibility of the formation of fusain by the bacterial decomposition of organic matter under alkaline anaerobic conditions is demonstrated experimentally. Sugars, starch, cellulose, wood, and beech leaves were allowed to decompose under soil containing sodium aluminosilicic complex. The decomposition products of the sugars and starch were entirely gaseous, being mainly methane and carbon dioxide, the latter being absorbed by the alkaline soil. Thus the presence of alkaline strata over coal seams may explain the absence of carbon dioxide from coal seam gases. The decomposition of cellulose does not yield a solid carbonaceous residue, but during peat formation it may give rise indirectly to "humic" substances which are decomposed under alkaline anaerobic conditions. The lignified cell walls of the beech leaves are, however, converted into a black material with the typical fusain structure, and as this artificial fusain resembles that found in peat under an alkaline roof in Egypt (B., 1926, 568) it may be concluded that bituminous coal has been produced by the decomposition of plant residues under alkaline anaerobic conditions.

H. D. Greenwood.

Improvement of coke by grinding the coal. Schweder (Gas- u. Wasserfach, 1927, **70**, 710—712).—A large-scale comparison of the coke produced from unground and ground coal shows that, in the latter case, the proportion of small coke and breeze is greatly reduced, the gas yield remaining unchanged. Suitable blending of the coals leads to a similar improvement in the coke. W. T. K. Braunholtz.

Properties and uses of coke. S. Qvarfort (Gas- u. Wasserfach, 1927, **70**, 646—651).—Coke for firing purposes should be strong and carefully graded to suit the various uses to which it is put, and its ash should have a high m.p. Chemical and physical properties of coke vary with the size of the coke, the more important factors being the content of ash and moisture, the weight per unit bulk volume, and the m.p. of the ash, all of which tend to increase with diminishing coke size, whilst there is a corresponding decrease in the calorific value. The formation of clinker from coke depends on the quantity and m.p. of the ash, the intensity of firing, the size of the coke, and its weight per unit bulk volume.

W. T. K. Braunholtz.

Removal of phenol from waste water from coking plants. F. Raschig (Z. angew. Chem., 1927, **40**, 897—898).—The phenol and pyridine contained in waste water from coking plants are extracted, before distilling off the ammonia, by treating the warmed water continuously with at least 30% of its volume of warm benzol in a special extraction tower. The benzol is then freed from all but 0·3% of phenol by distilling in a still with a column having Raschig rings and cooling dome of total height of 5 m. A. Cousen.

Lancashire coalfield. Ravine seam. II. Carbonisation in continuous vertical retorts. (Fuel Research Bd., Phys. and Chem. Survey of National Coal Resources, No. 9, 1927, 37 pp.).—Details are given of three large-scale carbonising tests (30—50 tons) carried out in Glover–West vertical retorts on coal from the Ravine seam (B., 1925, 305), using (1) 5% of steam for 120 hrs., (2) 5% of steam for 72 hrs., (3) 20% of steam for 120 hrs., the combustion chamber temperature averaging 1250—1260°. The coal contained 2·7% of moisture, 34·8% of volatile matter, and 11·2% of ash, and on carbonisation without steam gave a yield of 59·8 therms/ton. The yields/ton obtained with steaming were:—Test (1), gas (501 B.Th.U./cub. ft.) 69·1 therms, tar 12·6 gals., coke 13·4 cwt. Test (2), gas (488 B.Th.U.) 70·9 therms, tar 12·15 gals., coke 13·25 cwt. Test (3), gas (450 B.Th.U.) 85·3 therms, tar 12·75 gals., coke 12·8 cwt., showing practically no increase in tar yield, but an increase in gas yield of 23·4% when steaming is increased from 5 to 20%. The coke had the appearance of normal vertical-retort coke, but contained 14—18% of breeze. Its suitability for use in boilers and water-gas and suction-gas producers was examined by means of large-scale tests. Satisfactory results were obtained, but there was excessive clinker formation owing to the low fusion point of the ash (1280°). H. D. Greenwood.

Formation of naphthalene during high-temperature carbonisation. Y. Kosaka and Y. Oshima (Proc. Intern. Conf. Bituminous Coal, 1926, 463—468).—Cracking experiments at 850° indicate that the naphthalene of high-temperature tar is largely produced by decomposition and recombination of phenolic compounds. Chemical Abstracts.

Influence of oxygen, hydrogen, and nitrogen present in coke and of the admixture of distillation gases in the representation of producer gas by a gasification diagram. M. Ruhland (Gas- u. Wasserfach, 1927, **70**, 642—646, 664—667, 688—692, 712—716).—The relation between the analysis of producer gas, the details of its manufacture (*e.g.*, quantities of air and steam used, efficiency of gasification), and its properties (*e.g.*, calorific value, quantity of air required for combustion) can be represented graphically by a gasification diagram. The simplest case, in which pure carbon alone is gasified, *i.e.*, in which no methane is formed and all the oxygen, hydrogen, and nitrogen are derived from the air and steam ("primary gas"), may be represented by Ostwald's trapezoidal diagram or on a system of rectangular co-ordinates. Ordinary producer gas, of known composition, may be considered as comprising the "primary gas," together with methane and other gases derived from the oxygen, hydrogen, and nitrogen of the coke, and its properties may be deduced by the aid of the gasification diagram, or, more directly, knowing the analysis of the coke used, from a gasification equation for ordinary producer gas. Factors such as pressure, temperature, reaction velocity, etc. cannot be included in the gasification diagram. Numerous graphs and equations are given. W. T. K. Braunholtz.

Illuminating gas from lignite. K. Schmidt (Gas- u. Wasserfach, 1927, **70**, 657—661, 680—684).—Difficulties in the way of producing towns' gas from lignite include the powdery nature of lignite, its high water content

(50—55%), its low heat conductivity, the variability of its composition, and the high proportion of carbon dioxide evolved when it is carbonised. Of the German low-temperature carbonisation processes that of the Kohleveredlungs-A.-G. alone has found large-scale application for lignite. The crude gas obtained in this process has d 0·94, gross calorific value 5333, and contains roughly 29% CO_2, 12% CO, 16% H_2, 21% CH_4, 7% N_2, and 4% of heavy hydrocarbons (C_nH_m). The carbon dioxide is best removed by washing with water under pressure, whilst, in order to raise the hydrogen content and diminish that of heavy hydrocarbons, the gas could be subjected to subsequent cracking. Gas mixtures of roughly equal volumes of the low-temperature carbonisation gas with water-gas or with the gas obtained by further heating the lignite semi-coke to 1130° are very poisonous, owing to their high content of carbon monoxide (22—27%), but a mixture of 60% of the low-temperature gas and 40% of hydrogen has a composition, density, and calorific value approximating fairly closely to that of the usual mixture of coal gas and water-gas. Costs are given for the production of illuminating gas by this method. W. T. K. Braunholtz.

Effect of cooling on the naphthalene content of gas. E. Ott and F. Hinden (Gas- u. Wasserfach, 1927, **70**, 661—664).—The naphthalene content of the gas before the naphthalene scrubbers is decreased by cooling the gas. It is also reduced in the gas after the scrubbers, provided the naphthalene content of the anthracene oil in the final scrubber remains the same. By cooling the gas more gas can be passed through the scrubbers between two changes of oil. Rapid cooling is probably more effective than gradual cooling, as the tar fog formed will help to remove naphthalene, but cooling the gas below 23° before the naphthalene scrubbers may lead to a loss of heavy hydrocarbons.

W. T. K. Braunholtz.

Metaldehyde. C. Lew (Mon. pétrole roumain, 1925, **25**, 2002—2004).—A discussion of the production of metaldehyde, with a view to its use as a motor fuel, by the polymerisation of acetaldehyde below 0°.

Chemical Abstracts.

Effects of *X*-rays in photochemical oxidation, catalyst activation, and the ionisation of gaseous mixtures containing detonation inducers and suppressors. G. L. Clark (Brit. J. Radiol., Roentgen Soc. Sec., 1927, **23**, 112—124).—Irradiation with X-rays does not affect the rate of oxidation, and therefore the ageing, of rubber or oils. Irradiation with X-rays of the platinum catalyst in the manufacture of sulphuric acid produces an initial increase in activity, followed by a decrease to subnormal activity, an increase to normal activity, and a slow decrease. In a mixture of air and benzene irradiated with X-rays butyl nitrate (a knock inducer) slightly increased, and lead tetraethyl (a knock suppressor) markedly increased the ionisation; the action of such substances cannot therefore be attributed to decrease and increase, respectively, in the rate of recombination of ions. Chemical Abstracts.

Autoxidation of treated combustible liquids and its modification by other reagents, applied to "knock" in motors. C. Moureu, C. Dufraisse, and R. Chaux (Chim. et Ind., 1927, **18**, 3—12).—The authors consider that a spray of fuel in the liquid phase entering the cylinder of a motor forms peroxides, that the explosion of the latter is the cause of knock, and that anti-knock compounds impede and knock promoters favour the formation of peroxides. The experiments on which this theory is based were conducted with manometer tubes containing the fuel to be tested in a sealed bulb and immersed in an oil bath. The rise of the mercury in the manometer tube was a measure of the oxygen absorption. Curves showing the progressive rise of the mercury with the fuel alone and with 1·0% or 0·1% of various catalysts added enabled a comparison to be made. In this way it was shown that the absorption of oxygen by paraffin wax (b.p. 250—260°) was reduced by triphenylamine, lead tetraethyl, iodine, phenyl disulphide, and various other anti-knock compounds, but promoted by arsenic and thiourea. With some substances variations with time and dilution were observed. Other fuels studied were tetralin and decalin with generally similar results. Quinol, however, acted as an oxidation promoter for the first and retarder for the second. A sample of petrol showed only very slight oxygen absorption, although redistilled fractions gave much higher figures. These fractions, after oxidation, gave a coloration with potassium iodide. Bromine and nitric acid, well known knock promoters, promoted oxygen absorption by the higher-boiling fraction of the petrol at 160°; others such as sodium nitrate were inert. It was reduced by lead tetraethyl, phenyl disulphide, sulphur, iodine, etc. Marine animal and vegetable oils also show varying amounts of oxygen absoption at 160°. C. Irwin.

Factors affecting internal combustion. C. I. Kelly (J. Inst. Petrol. Tech., 1927, **13**, 101—123).—The history of the internal-combustion engine and the phenomena of flame propagation and detonation are reviewed. The pulsatory nature of the flame wave is shown in photographic reproductions. H. Moore.

Origin of petroleum: Berginisation of fish-liver oils and other materials. W. R. Ormandy, E. C. Craven, I. M. Heilbron, and H. J. Channon (J. Inst. Petrol. Tech., 1927, **13**, 1—20).—The literature by Tsujimoto, Chapman, and others on the hydrocarbon oil (squalene) present in the liver oils of the *Squalidæ* sharks is reviewed, and its possible connexion with the origin of petroleum is indicated. Samples of squalene having d^{20} 0·8559 and ignition temperature (Moore) 259° were Berginised in a small bomb. The resultant liquid had d^{20} 0·8256, and yielded on distillation 57% of spirit up to 200°, 23% of kerosene at 200—300°, and residual heavy oil. The spirit fraction contained 10% of unsaturated, 26% of aromatic compounds, 24% of open- and 40% of closed-chain paraffins. In a further Berginisation test at 470° for 1 hr. with initial pressure of hydrogen of 80 atm., falling to 63 atm. on cooling, the liquid yield was 75%, the absorption of hydrogen 3·45%, and the product had d^{20} 0·8129 and yielded 59·3% of spirit up to 180° and 28·2% of kerosene up to 300°, no wax being found in the residue. It contained 6·8% of unsaturated and 17·9% of aromatic compounds and 75·3% of paraffins. The presence of *iso*pentane was well marked. A sample of d-pinene when Berginised

gave very similar results to squalene, as was the case with some lignite oils, but the latter yield some 20% of phenols. The higher phenols when Berginised are converted into paraffins and lower phenols; the lower the phenol the more soluble it is in water.

H. Moore.

Extraction of amorphous wax from laboratory specimens of oil [petroleum]. S. Bowrey (J.S.C.I., 1927, **46**, 228 T).—The large-scale centrifugal process for the separation of amorphous paraffin wax from oil, after the addition of petroleum naphtha, is imitated in the laboratory in a short time by cold-settling in a vacuum flask, the jacket of which is half filled with liquid sulphur dioxide. By evaporating the latter under a partial vacuum the oil mixture can be cooled at any desired rate without causing sufficient convection to hinder the settling of the wax. Settling at — 20° F. produces an oil of cold test 25—30° F. W. J. Powell.

Pipe still for continuous distillation in the laboratory. A. A. Ashworth (J. Inst. Petrol. Tech., 1927, **13**, 91—100).—The temperature at which light oils are vaporised in a pipe still is 80—100° lower than that in usual continuous crude stills. In the laboratory pipe still the crude oil is fed at 7—10 c.c./min. into an inclined tube of ⅝ in. internal diam., through which passes an electric heater consisting of a coil of nichrome wire wrapped round a tube of ¼ in. outside diam. A temperature of about 300° is attainable. The oil vapours from the inclined tube enter a chamber filled with glass beads nearly up to the level of a thermometer which registers the vapour temperature. The inclined tube and chamber are heat-insulated. A thermometer in reversed position enters the bottom of the chamber, and indicates the oil temperature, and the residue leaves the chamber at this point and passes through a cooler to a receiver, while the vapours from the chamber leave at the top, passing through a condenser to a receiver. The vapours are kept in intimate contact with the oil, the temperatures being measured immediately after the separation of vapour and oil. The temperatures shown for percentage of distillate, though starting at the same initial point, are considerably lower than those in a flask distillation, as shown in graphs. The variations in temperature may be kept within 2°. The still may be used for observing the equilibrium temperatures of light distillates. H. Moore.

Corrosive influence of sulphur and sulphur compounds in naphtha solutions. H. Schmidt (Petroleum, 1927, **23**, 646—648).—Strips of aluminium, zinc, steel, copper, brass, and silver were immersed in a closed test tube at 50° for 3 hrs. in a petroleum fraction of d^{20} 0·769, flash point 41°, initial b.p. 151°, containing 0·010—0·360% of sulphur in the form of *iso*amyl mercaptan, hydrogen sulphide, carbon disulphide, methyl sulphate, *p*-toluenesulphonic acid, butyl sulphide, thiophen, etc. Iron and aluminium were not attacked, zinc and brass were feebly corroded, copper and silver were energetically attacked by free sulphur. In five-day tests with the addition of water, aluminium showed corrosion in many cases. In trials in a copper dish free sulphur, carbon disulphide, and mercaptan gave deposits of copper sulphide. In thirty-day tests at room temperature chromium and zinc were most resistant; most metals were attacked, and especially manganese. Mercaptans had the greatest corrosive influence. The corrosive action of the sulphur compounds corresponded to their capacity for ionisation. Heat promoted corrosion.

H. Moore.

Methods of testing oils. I. Viscosity determinations with the Engler apparatus. H. Schlüter (Chem.-Ztg., 1927, **51**, 565—566).—In the usual Engler test the thermometer shows a slow fall in temperature as the oil flows through the capillary owing to the gradual exposure of the bulb of the thermometer. This error may be avoided by using a thermometer with the bulb bent round at right angles to the stem, so that it lies in a horizontal position just above the floor of the oil container. A second thermometer with the bulb in the normal position serves as a means of closing the capillary entrance before and after the desired amount of oil has flowed out. When working at 50° the temperature of the water bath should not exceed 50·3° for maintaining the oil at a constant temperature of 50°. A. R. Powell.

Aerosols. Beyersdorfer.—See I.

Behaviour of cellulose on heating. Bain and others. —See V.

Phenols from ammonium sulphate still effluents. Parkes.—See VII.

Chequer bricks for oil-gas manufacture. Knollman.—See VIII.

Penetrance of oily fluids in wood. Howald.— See IX.

Patents.

Treatment of pulverised vegetable fuels. S. Shimamoto (E.P. 273,556, 30.10.26).—Sawdust or other vegetable matter is steeped in hot slaked lime until the tissues are soft and sticky, powdered coal is added, and the mixture briquetted. The dry briquettes may be allowed to absorb oil or other liquid fuel.

A. B. Manning.

Drying apparatus for briquettes and other agglomerates. F. M. Crossman (E.P. 267,784, 4.8.26). —A vertical shaft carrying a series of radial arms rotates in a vertical drum consisting of a number of superimposed chambers. Each chamber has a floor opening, the several openings being staggered with respect to one another. The drum has its curved surface perforated, and is surrounded by a hot chamber provided with means for supplying hot furnace gases to and withdrawing them from the drum. Briquettes to be dried are fed into the uppermost compartment of the drum, from whence they are transferred by the action of the rotor to successively lower compartments. The briquettes are dried by contact with the hot furnace gases. S. Pexton.

Froth-flotation concentration of coal. P. T. Williams, and Minerals Separation Ltd. (E.P. 272,301, 12.3.26).—A salt of an organic derivative of a thiocarbonic acid (*e.g.*, an alkali xanthate) is used as a flotation reagent. It may be admixed with the coal prior to charging it to the separator, or used with a soluble and readily disseminable frothing agent. Use

of an organic protective colloid (*e.g.*, starch) is suggested to retard the flotation of high-ash fractions; the flotation circuit may also be rendered alkaline by lime, which, by flocculating gangue-slimes, assists the separation of water from the tailings. R. A. A. Taylor.

Coke ovens. H. Schröder (E.P. 266,996, 20.8.26).—A separately controlled gas feed is provided for the end pair of heating flues in an oven with vertical flues. The regenerators are built in two horizontal sections, so that the air which enters the battery from outside reverses its passage in the upper half of each regenerator and passes to the combustion chambers through a conduit immediately adjacent to the end walls of the battery and enters the distribution flue immediately below the ports for supplying the end two combustion flues. The main damper controlling the secondary air is disposed in the distributing flue beyond the nostrils to the end combustion flues, in such a way that there is always a relatively larger proportion of air passing to each of the end combustion flues than to the remainder. Radiation losses from the end walls are therefore neutralised by burning an increased proportion of gas in the end two flues. S. Pexton.

Coking or carbonising ovens. Stettiner Chamotte-Fabrik A.-G. vorm. Didier (E.P. 263,801, 21.12.26. Conv., 24.12.25).—An inclined carbonising oven has its sole so inclined that the discharging mechanism can be mounted on the carriage of the charging truck. The producer, which is built into the setting, can be charged from the same stage as the ovens. S. Pexton.

Gasification of caking coals in a gas producer. K. Koller (E.P. 257,633, 31.8.26. Conv., 31.8.25).—Within the upper part of, and in the axis of, a gas producer is arranged a water-cooled distillation bell which can be rotated. The section of the bell is not circular, and therefore the expanding coal undergoing caking is sheared against the stationary mass of fuel undergoing gasification in the main part of the producer. S. Pexton.

Distillation or heat treatment of carbonaceous or like materials. Salermo, Ltd., and E. M. Salerni (E.P. 273,528, 15.9.26. Cf. E.P. 247,300; B., 1926, 308).—The material, crushed to pass a $\frac{3}{8}$-in. sieve, is conveyed in a thin layer over flat metal plates in a drying chamber above the distillation chamber, into which it then passes through a gas-tight seal. In the distillation chamber it passes laterally through a series of horizontal, semi-cylindrical troughs arranged side by side, each trough being provided with a combined scraper and stirrer. The spent material is discharged into a coke-cooling chamber or may be fed directly to the furnace of a steam generator. The combustion chamber is divided from the distillation chamber below by a series of brickwork arches which leave a channel between the two chambers through which the hot combustion gases flow in a direction opposed to the motion of the material in the retort. The hot gases flow thence to the drying chamber, through which they again pass counter-current to the material. The products of distillation pass from the retort through a dust extractor to a condenser. The temperature of carbonisation is 500—550°; an installation consisting of a single retort with 12 troughs each 10 ft. long and 8 in. radius can deal with 75 tons of coal per 24 hrs. A. B. Manning.

Treatment of coal. Internat. Combustion Engineering Corp., Assees. of W. Runge and E. A. Packhard (E.P. 249,086, 4.2.26. Conv., 12.3.25).—Coal is pulverised in the presence of a hot gaseous medium and the mixture is conducted to a cyclone separator from which the powdered fuel is transferred to the distributing hopper of a carbonising unit. The hot gas, usually air, is utilised for burning part of the purified gas yielded by carbonisation in order to preheat the air to the pulveriser. The remaining gas from carbonisation may be burnt in the retort down which the powdered fuel to be carbonised is showered, or it may be used to preheat some gaseous distillation medium supplied to the retort. S. Pexton.

Vertical retort settings for the destructive distillation of coal and the like. F. J. West, E. West, and West's Gas Improvement Co., Ltd. (E.P. 273,474, 18.6.26).—A large volume of gas of low calorific value is produced by steaming in vertical retorts, the gas being raised to the desired calorific value by admixture with oil-gas from a generator which forms part of the vertical-retort setting, and is heated by the waste gases therefrom. The oil-gas generators may be of the vertical or horizontal type, and are provided with doors through which the carbon or other deposit may be periodically removed. A. B. Manning.

Method and apparatus [electric furnace] for treatment of carbonaceous material. J. J. Naugle (E.P. 267,240—1, 14.12.25).—(A) Comminuted carbonaceous material, *e.g.*, leached carbonised lignin residue derived in the production of wood pulp, is washed with acidified water and filter-pressed. The product is carbonised in an electric furnace with or without the addition of sodium fluoride or fluxes which render the silica volatile and slag the impurities respectively. Further purification is effected by allowing the impurities to settle from a suspension of the carbonised product in water. The carbon has a high electrical conductivity and a high absorptive power. The furnace consists of a rotary kiln carrying mixing members from its internal periphery. The mixing members are insulated from the casing and support the electrodes for heating. Material under treatment is fed from a hopper to a spiral device which ensures long and uniform heating. The necessary oxidising medium (steam or air) facilitates transference of the material to the outlet. (B) The electric furnace is a horizontal cylindrical casing carrying a central revolving electrode built up in sections to enable the heating to be carried out in stages of varying intensity; *e.g.*, the material may be heated at 1500—800° in the first stage, 800—600° in the second stage, and 600—400° in the third stage. S. Pexton.

Producing high-grade combustible gases from bituminous fuel. A. H. Lymn (E.P. 271,173, 23.2.26).—The apparatus comprises a combined generator and retort together with an external retort. The retort superimposed on the generator is heated internally by the "up-run" gases admixed, if necessary, with super-

heated steam. The separate retort is heated externally by the blow gases from the generator after admixture with air and internally by the "down-run" gases. The distillation gases from both retorts pass to a common seal pot. In one design the coke from the separate retort is fed to the hopper of the combined retort and generator. The temperature of carbonisation of the separate retort is controlled by admixing cool gases with the hot blow gases. A. C. Monkhouse.

Drying of fuel gas. C. Cooper, D. M. Henshaw, and W. C. Holmes & Co., Ltd. (E.P. 266,825, 5.12.25).—Hot producer, generator, or blast-furnace gas is washed with a solution of a hygroscopic salt immediately before its combustion in a furnace. The process efficiently removes mechanically carried water and can be conducted without reducing the temperature of the gas. The absorbed water is evaporated from the desiccating solution by spraying it over a series of steam-heated pipes. S. Pexton.

Manufacture of hydrocarbons and derivatives thereof from natural oils and other bitumens. I. G. Farbenind. A.-G. (E.P. 249,493 and 273,228, 26.2.26. Conv., [A], 19.3.25, [B], 26.2.26. Addns. [A, B] to E.P. 247,584; B., 1927, 595).—Modifications of the process described in the main patent are claimed whereby, instead of solid carbonaceous materials, (A) mineral oils or other bitumens or distillation or extraction products thereof, or (B) conversion products or hydrogenation products of mineral oils or bitumens, are employed. A. B. Manning.

Manufacture of liquid or other hydrocarbons and derivatives thereof from coal, tars, mineral oils, etc. I. G. Farbenind. A.-G. (E.P. 251,264, 14.4.26. Conv., 25.4.25. Cf. E.P. 247,583 (B., 1927, 595), 247,586, 249,501).—Tars, oils, etc. are treated with hydrogen or reducing gases containing combined hydrogen at a high temperature (450—550°), under a pressure of at least 50 atm., in the presence of contact masses containing tungsten, chromium, or their compounds. By this process, for example, cresols may be reduced to aromatic and hydroaromatic hydrocarbons, and high-boiling oils cracked to produce a high percentage of benzines without any coke formation. A. B. Manning.

Refining and desulphuring light oils, especially low-temperature benzines. A. Riebeck'sche Montanwerke A.-G. (G.P. 439,608, 11.6.25).—Aldehydes, aldehyde polymerides, or substances which produce aldehyde in the presence of polymerising agents such as acids or alkalis, are used as refining agents; the impurities containing sulphur are precipitated in an insoluble form and can be separated from the oil. A. B. Manning.

Purification of low-temperature benzines. Zeche M. Stinnes, Assees. of F. Müller and P. Hützen (G.P. 439,006, 8.11.23. Addn. to G.P. 437,048; B., 1927, 468).—The procedure of the main patent is modified by the use of acetone in place of alcohol as washing liquid. The purified benzine is equal in quality to that obtained when alcohol is used. A. B. Manning.

Apparatus for cracking mineral oils. Werschen-Weissenfelser Braunkohlen A.-G., and A. Fürth (G.P. 439,010, 12.12.22).—In the method of cracking oils, especially high-boiling paraffin hydrocarbons, by pressure distillation with a catalyst in the vapour space, the higher-boiling condensate being refluxed in such a manner that it does not come into contact with the catalyst, the latter is arranged in an annular space between two cylinders in a decomposition chamber affixed to an autoclave. A. B. Manning.

Continuous distillation of mineral oils etc. E. Blümner (G.P. 439,712, 31.7.21. Addn. to G.P. 338,846; B., 1922, 496 A).—The liquid mixed with a gas, *e.g.*, hydrogen, is passed in a finely-divided form through an externally heated bath of molten metal, and so brought into contact with catalysts contained as a finely-divided filling or other suitable form in the melt. The process is especially suitable for bringing about chemical reactions between liquids and vapours or gases. A. B. Manning.

Production of paraffin hydrocarbons with more than one carbon atom. F. Fischer and H. Tropsch (E.P. 255,818, 26.3.26. Conv., 21.7.25).—Oxides of carbon, mixed with hydrogen, are reduced to paraffins higher than methane by heating the gases at about the ordinary pressure in presence of a catalyst at temperatures between that at which the catalyst begins to act and that at which methane is the main product. The temperature employed is generally 80—100° below that at which methane only is formed. *E.g.*, using as catalyst a mixture of zinc oxide and finely-divided iron, at 300° the product contains 10% of methane and 90% of higher paraffins, whilst at 430° methane is the only hydrocarbon. A. Davidson.

Improving mineral oils and tar products. Siemens & Halske A.-G. (E.P. 263,186, 17.12.26. Conv., 19.12.25).—Oils or tar products are freed from easily oxidisable constituents by treatment with ozone in the presence of inorganic adsorbents, *e.g.*, silica gel or iron oxide, preferably mixed with a basic substance such as calcium oxide. Fatty acids may be recovered from the adsorbing material by subsequent treatment with steam. A. B. Manning.

Production of lubricants containing water. M. J. Heitmann (E.P. 250,562, 29.3.26. Conv., 11.4.25).—Emulsified lubricating oils containing water are prepared by passing saturated or superheated steam, or a gas, under pressure into the lubricating oil, and then adding the requisite amount of water through distributing nozzles to the finely divided oil, continuing the passage of steam or gas until a stable emulsion is produced. Emulsified solid greases are similarly produced by adding materials containing higher alcohols, or other agents for increasing viscosity, to the lubricating oil before proceeding as above. A. B. Manning.

Filtering apparatus [for lubricating oil]. C. S. Garland, T. E. Beacham, and J. A. Pickard (E.P. 273,364, 1.3.26, 8.4.26, and 15.4.26).—The patent deals with the construction and methods of heating edge filters for cleaning the lubricating oil of internal-combustion engines. The filtering medium may be made of commercial asbestos sheets purified by incineration after assembly. B. M. Venables.

Motor fuel. H. Herzog and W. Hüssy-Bühler (G.P. 439,550, 11.3.26).—A 9 : 1 mixture of crude or

illuminating petroleum and alcohol, either pure or containing water, is passed under pressure and at higher temperatures over or through a catalyst (*e.g.*, molten lead, to which bismuth or cadmium has been added to lower the m.p.), and the resulting liquid, after removal of the solid hydrocarbons, mixed with 20—50% of benzine.

A. B. MANNING.

Obtaining light hydrocarbons from heavy cyclic hydrocarbons or derivatives. A. J. KLING and J. M. F. D. FLORENTIN (E.P. 253,507, 26.5.26. Conv., 12.6.25).—See F.P. 607,155; B., 1927, 548.

Coal pulverisers. A. S. CACHEMAILLE. From S. A. S. BUNTING (E.P. 274,278, 20.7.26).

Screen for separating or sorting coals, minerals, etc. H. LOWSON, and H. LOWSON, LTD. (E.P. 274,582, 26.1.27).

Gasworks retort settings. H. J. TOOGOOD, and R. DEMPSTER & SONS, LTD. (E.P. 274,154, 15.3.26).

Powdered fuel furnaces. A. B. HELBIG (E.P. 266,827, 5.12.25).

Separation of gases (E.P. 267,369).—See I.

Acetic acid (F.P. 601,156).—See VII.

Carbon disulphide (G.P. 439,766).—See VII.

III.—TAR AND TAR PRODUCTS.

Basic nitrogen compounds from Fushun shale tar. T. EGUCHI (Bull. Chem. Soc. Japan, 1927, **2**, 176—186).—The tar obtained by distilling Fushun shale in vertical Oakbank retorts contains 2—4% of bases. The fraction of the crude tar of b.p. 220—250° yields bases containing no primary or secondary amines, and only traces of pyrroles. The following pyridine derivatives (cf. Garrett and Smythe, J.C.S., 1903, **83**, 763; Oshima and Ishibashi, B., 1926, 1006) were isolated by repeated fractional distillation, purified through the mercurichlorides, and identified by oxidation to the corresponding carboxylic acids: 2-methyl-, n_D^{25} 1·4983; 2:6-dimethyl-n_D^{25} 1·4953; 2:4-dimethyl-, n_D^{25} 1·4984; 2-methyl-6-ethyl-, b.p., 160—161°; d_4^{25} 0·9207, n_D^{25} 1·4950; 2:4:6-trimethyl-, d_4^{25} 0·9101, n_D^{25} 1·4959. Displacement of a methyl group from the 4- to the 2-position increases the mol. refraction by 0·13 to 0·18.

H. E. F. NOTTON.

Phenol from coking plants. RASCHIG.—See II.

Phenols from ammonium sulphate still effluents. PARKES.—See VII.

PATENTS.

Decomposition of tars or tar oils into pitch and oil without distillation. H. WAIBEL (G.P. 439,804, 1.7.26).—The tar is treated with a non-aromatic organic solvent subsequently to being heated in a vessel provided with a reflux condenser. Steam may be passed through the tar while it is being refluxed, any tar oils in the escaping vapours being recovered in a known manner. The oils obtained do not thicken on keeping in the air.

A. B. MANNING.

Fractional distillation. V. FREUND (Austr. P. 104,137, 6.5.24).—To separate liquid mixtures into their constituents of definite b.p., as for example tar light-oils into benzene, toluene, and xylene, the mixed vapours issuing from the distillation column are lead through dephlegmators provided with reflux condensers in which the cooling liquids used have b.p. coinciding with those of the respective fractions to be condensed.

A. B. MANNING.

Production of benzene by reaction between phenolic vapours and a reducing gas. A. IRINYI (E.P. 273,493, 7.7.27).—In the reduction of phenol to benzene at 750° by hydrogen or gases containing it, efficient temperature control is obtained by separately heating the phenol vapours and the reducing gases to the reaction temperature before mixing. Each component is heated by passing part of the gas or vapour through a superheater in which it is raised above the reaction temperature, by-passing the remainder, and mixing the superheated part with the remainder as required to obtain the necessary temperature. A. DAVIDSON.

Hydrocarbons etc. (E.P. 251,264).—See II.

Tar products (E.P. 263,186).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Cold oxidation (decolorisation) of colouring matters by sodium hypochlorite in acid solution. II. Oxidation of azo dyes. A. SEYEWETZ and E. CHAISE (Bull. Soc. chim., 1927, [iv], **41**, 332—351; cf. A., 1927, 353).—The decolorising (oxidising) action of sodium hypochlorite on 2% aqueous solutions or suspensions of a number of representative azo dyestuffs in the presence of hydrochloric acid has been studied at 0—5°. Azo compounds, such as azobenzene or azotoluene, which contain no auxochromic group are not attacked under these conditions. All other azo compounds examined are decomposed at the azo group nearest to the auxochrome group, affording the original diazonium compound together with an oxidation or chlorination product of the amine or phenol residue. Thus aminoazobenzene affords benzenediazonium chloride together with a tarry product containing chloranil. Orange III is more readily attacked, and about 70% is converted into diazobenzene-*p*-sulphonic acid, dimethylamine, benzoquinone, and chloranil. With Orange IV oxidation of the diphenylamine occurs, and only traces of diphenyl were isolated besides the diazobenzene-*p*-sulphonic acid. Dyes such as Chrysoidine CR, containing a *m*-diamine group, probably afford, in addition to the diazo compound, an amiuoquinone or a triazole derivative. Dyes containing a phenolic group afford a chlorinated quinone in addition to the diazonium salt. Thus Tropæoline Y affords 60% of the diazobenzene-*p*-sulphonic acid and chloranil whilst Chrysoine yields the same diazo compound and an unidentified chlorinated hydroxyquinone. Orange I and Orange II behave similarly, the diazobenzene-*p*-sulphonic acid being accompanied by 2-chloro-1:4-naphthaquinone in the former case and 3:4-dichloro-3:4-dihydro-1:2-naphthaquinone, m.p. 86°, together with a little *iso*naphthazarin, m.p. 275°, from oxidation of the dichlorodihydronaphthaquinone in the latter case. Side-chains are not attacked, Orange GT affording *p*-toluenediazonium chloride, whilst the presence of sulphonic groups in the phenol or naphthol nucleus leads to the formation of sulphonated quinones. Thus Ponceau 6R crystals affords α-naphthalene-

diazonium chloride and 1 : 2-naphthaquinone-6 : 8-disulphonic acid. Mordant azo dyestuffs behave normally; Anthracene Yellow RN, for instance, yields *p*-nitrobenzenediazonium chloride andtrichloro-*p*-benzoquinonecarboxylic acid, m.p. 61—62°. Brilliant Croceine M and Naphthol Black WB are typical of the behaviour of polyazo colours, the former affording azobenzene-*p*-diazonium chloride and 1 : 2-naphthaquinone-6 : 8-disulphonic acid, and the latter benzenediazonium chloride, *p*-nitrobenzenediazonium chloride, and (probably) 1 : 2 : 7 : 8-naphthadiquinone-4 : 6-disulphonic acid. Polyazo dyestuffs derived from diamines behave analogously to the monoazo colours. Thus Congo Red affords diphenyl-4 : 4′-tetrazonium chloride and 1 : 2-naphthaquinone-4-sulphonic acid, Chrysamine K yields the same tetrazo compound and trichlorobenzoquinonecarboxylic acid, m.p. 61—62°. Brilliant Yellow F affords benzoquinone, chloranil, and the tetrazo derivative of diaminostilbenedisulphonic acid, but the corresponding dihydroxystilbenedisulphonic acid could not be obtained from the tetrazo compound on account of its instability. With carbamide derivatives, such as solid Diamine Yellow 3G (Cassella), carbon dioxide is first liberated, with formation of a hydrazine derivative, which is then oxidised to the corresponding azo derivative. The original azo groups are attacked in the usual way with formation of the diazonium salt and production of the chlorinated quinone. Pyrazolone derivatives such as Tartrazine afford the diazonium salt and the pyrazolone, the latter resisting oxidation. 1-*p*-Sulphophenyl-5-pyrazolone-3-carboxylic acid is not oxidised under these conditions, but loses carbon dioxide. R. BRIGHTMAN.

Coloration of beeswax. JAUBERT.—See XII.

Determination of methylhexalin. LINDNER and ZICKERMANN.—See XX.

PATENTS.

Production of azo dyes and lakes insoluble in water. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 250,909, 19.2.26. Conv., 18.4.25).—Azo pigments suitable for lakes and for dyeing rubber are obtained by coupling 2 : 3-hydroxynaphthoic β-naphthylamide with a diazotised arylamine containing substituents (at least one being a chlorine atom) in positions 2 and 5 (cf. E.P. 235,169; B., 1925, 666, for corresponding ice-colours). Examples of diazo components are 4-chloro-*m*-toluidine (red), 4-chloro-*o*-toluidine (bluish-red), 4-chloro-*o*-anisidine (bordeaux), 4-chloro-*m*-anisidine (red), 4-chloro-*o*-phenetidine (bluish-bordeaux), 2 : 5-dichloroaniline (red), 4-chloro-2-aminodiphenyl ether (bluish-red), 4-chloro-2-aminophenyl benzyl ether (bluish-red). C. HOLLINS.

Intermediate products for dyes. H. FRITZSCHE, E. KRUMMENACHER, H. GUBLER, and O. KAISER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,625,530—3, 19.4.27. Appl., 20.7.23. Conv., 7.9.22).—See E.P. 221,843; B., 1924, 937. The *products* obtained by the condensation of cyanuric chloride (1 mol.) with *m*-aminobenzoic acid (1 mol.), and with *p*-aminosalicylic acid (1 mol.), have m.p. 220° and above 360°, respectively. T. S. WHEELER.

Preparation of indanthronedisulphonic acid and indanthrone. BRITISH DYESTUFFS CORP., LTD., A. SHEPHERDSON, and A. J. HAILWOOD (E.P. 274,226, 26.4.26).—1-Nitroanthraquinone-8-sulphonic acid is reduced in neutral or alkaline solution, *e.g.*, with zinc dust and ammonium chloride or sodium sulphide insufficient for complete reduction of the nitro-group, giving 1-*hydroxylaminoanthraquinone*-8-*sulphonic acid*, which when boiled with caustic alkali is converted into indanthronedisulphonic acid, a greenish-blue acid dye. Electrolytic desulphonation of the product yields leuco-indanthrone. C. HOLLINS.

Dyes and dyeing. [Sulphuric esters of leuco-vat dyes.] H. A. E. DRESCHER, J. E. G. HARRIS, B. WYLAM, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 274,156, 15.3.26).—A vat dye is reduced with the aid of a metal (zinc, copper) to its leuco-derivative in presence of a tertiary base (pyridine) and an alkyl halide or a quaternary ammonium halide, together with the sulphur trioxide derivative of the tertiary base (made, *e.g.*, by treating pyridine with chlorosulphonic acid, an alkyl chlorosulphonate, oleum, or sulphur trioxide). The soluble sulphuric ester of the leuco-vat dye is isolated and applied in the usual manner. The vat dyes specified in the examples are indigo, thioindigo, and dimethoxydibenzanthrone. C. HOLLINS.

Dyes and dyeing. [Sulphuric esters of leuco-vat dyes.] B. WYLAM, J. E. G. HARRIS, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 274,303, 1.3.26; cf. E.P. 247,787 and 260,638; B., 1926, 403; 1927, 39).—Leucoflavanthrone is treated with chlorosulphonic acid in presence of a tertiary base (pyridine) at 45°. Addition of alkali (ammonia solution) precipitates a reddish derivative, which is purified by solution in dilute aqueous sodium carbonate and aeration; from the filtrate the reddish-yellow compound may be salted out, and used directly for dyeing and printing without vatting, the colour being developed by hydrolysis and oxidation. C. HOLLINS.

Greenish-blue to green vat dyestuffs of the thioindigo series. R. HERZ and J. MÜLLER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,623,410, 5.4.27. Appl., 23.3.26. Conv., 25.10.24).—2-Amino*peri*naphthoxypenthiophens are condensed with substituted or unsubstituted 3-oxythionaphthens, to give thioindigo vat dyes, which yield fast greenish-blue to green shades on wool and cotton. 2-Acylamido*peri*naphthoxypenthiophens can also be employed, the acylamido-group being hydrolysed before or after condensation. *E.g.*, 2-acetamido*peri*naphthoxypenthiophen (cf. U.S.P. 1,498,913; B., 1924, 781) is hydrolysed with aqueous-alcoholic hydrogen chloride, and the product is condensed in boiling glacial acetic acid containing sodium acetate with 6-chloro-2-(*p*-dimethylaminoanilo)-4 : 5-benzo-3-oxythionaphthen to yield a *dye*, which gives strong yellowish-green shades on cotton and wool in a hyposulphite vat. T. S. WHEELER.

Preparation of perylenequinones. COMP. NAT. MAT. COL. ET MANUF. PROD. CHIM. DU NORD RÉUNIS, ÉTABL. KUHLMANN, and H. PEREIRA (F.P. 592,272, 13.1.25. Conv., 16.4.24).—Halogenated perylenes are oxidised with hot concentrated sulphuric acid. Dibromoperylene of m.p. 278° when heated with sulphuric acid loses bromine and hydrogen bromide at 95—100°;

after being heated to 130—140° and poured into water it gives a *product*, m.p. above 360°. Dibromoperylene of m.p. 189° in similar manner yields 3 : 10-perylenequinone.

C. HOLLINS.

Preparation of vat dyes and intermediates from diacylperylenes. COMP. NAT. MAT. COL. ET MANUF. PROD. CHIM. DU NORD RÉUNIS, ÉTABL. KUHLMANN (F.P. 612,338, 5.3.26).—Dibenzoylperylene is oxidised in 100% sulphuric acid with manganese sulphate or dioxide at 10—15° in presence of boric acid to a red-violet vat *dye*, which is improved in fastness by chlorination in nitrobenzene at 60°. With less oxidising agent the *product* dyes cotton in olive shades changing to blue on acidification, and may be methylated to a fast bluish-green vat dye. Ditoluoylperylene behaves similarly.

C. HOLLINS.

Preparation of vat dyes of the perylene series. F. BENSA (Austr. P. 104,385, 3.11.24).—Dihalogenated diacylperylenes are heated with alkalis or alkaline earths in presence or absence of an organic solvent (aniline, quinoline, etc.). Dibromo- or dichloro-dibenzoylperylene, obtainable from dihalogenoperylenes, benzoyl chloride, and aluminium chloride, gives fast violet vat *dyes*. *Dichlorodi-α-chlorobenzoylperylene*, m.p. 355°, gives a violet-blue; *dichlorodi*-p-*toluoylperylene*, m.p. 325°, a violet; *dichlorodi*-m-*toluoylperylene*, m.p. 334°, a violet; *dichlorodi*-o-*toluoylperylene*, m.p. 351°, a blue; *dichlorodi-α-naphthoylperylene*, m.p. 275°, a blue. C. HOLLINS.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Use of hydrocyanic acid gas for the fumigation of American cotton on import into India. A. J. TURNER and D. L. SEN (Agric. J. India, 1927, **22**, 173—175).—Hydrocyanic acid gas can be used successfully to destroy the Mexican boll weevil (*Anthonomus grandis*) in baled cotton. Cotton absorbs hydrocyanic acid whether it be loose or baled, damp or dry, but there is no evidence of any chemical combination. The moisture content of the cotton affects the extent of the absorption of gas, but temperature has little effect. In generating gas, sodium cyanide in an amount equal to 0·05% of the weight of cotton is sufficient in a gas-tight vessel. In barge-holds, where there is some leakage, 0·075% of the weight of cotton is recommended. In laboratory experiments the best results were obtained with a gas concentration of 450 parts per 100,000 for 6 hrs., followed by a period of 14 hrs. at a concentration of 200 parts per 100,000. A. G. POLLARD.

Decomposition of straw and nutritive value of the decomposed straw. F. HONCAMP (Cellulosechem., 1927, **8**, 81—91).—Of the factors involved in the valuation of vegetable foodstuffs, only the nitrogen-free extract and the "crude fibre" are of importance from the point of view of the decomposition of straw. By "crude fibre" is understood all the nitrogen-free substances undissolved after heating with 1¼% sulphuric acid and potassium hydroxide for ½ hr., and washing with water, alcohol, and ether. The nitrogen-free extract comprises all the organic plant substances insoluble in ether, and not belonging to the nitrogenous and ash constituents, which, however, dissolve in the reagents used in the determination of crude fibre. This extract is a complicated mixture of carbohydrates (including sugars and pentosans), substances not belonging to the carbohydrate groups (*e.g.*, organic acids, lignins, aromatic substances, etc.), and unchanged and modified cellulose. Similarly, the crude fibre includes cellulose, pentosans, and incrusting substances such as lignin. These two groups form about 90% of the material of all the straws examined, including grain, rape, beet, and leguminous straws (nitrogen-free extract 33—45%, and crude fibre 42—57% by wt. of dry material). Only one sixth to one seventh of the total pentosan is contained in the crude fibre, the remainder being in the nitrogen-free extract. For various straws examined, the crude fibre was found to contain 64—81% of pure cellulose, 2—3% of cutin, and 16—32% of lignin. The digestible part of the crude fibre has the same percentage composition as pure cellulose, the following mean values being given by three different methods for a number of different straws C 44·70, 44·46, and 44·11%, and H 7·22, 6·49, and 6·63% (pure cellulose $C_6H_{10}O_5$ requires C 44·4%, H 6·2%, and O 49·4%); the somewhat higher values for hydrogen are attributed to the hydrating action of the reagents used. Further, for the ash and protein-free digestible part a calorific value of 4219·6 was found, that for pure cellulose being 4185·4. Hence there appears no doubt that the cellulose of the crude fibre is to a considerable extent decomposed and absorbed in the intestinal canal of animals. The pentosan also present in the digestible part of the crude fibre appears to have the same calorific value as the carbohydrates, starch, and sugar. Experiments show that cellulose is the substance which, in ruminants, is split up by fission fungi into carbon dioxide, methane, acetaldehyde, acetic and butyric acids. The digestible crude fibre, freed from incrusting substances, possesses a nutritive value similar to that of the digestible part of starch flour. The smaller food value of straws is conditioned not by the inferior nature of their nutritive material, but by the fact that the latter is covered with incrusting substances which permit only limited access to the digestive juice or intestinal bacteria, a higher value being obtained when these are removed or loosened. The latter is achieved mechanically by finely grinding the straw, or better, by treatment of the material with caustic alkalis with or without heat and increased pressure. Decomposition with hydrochloric acid is unsuitable. By use of 4% sodium hydroxide in an autoclave, the digestibility of wheat chaff was increased from 26 to 56%, and of the crude fibre from 37 to 83%. The extent and nature of the decomposition depend upon the temperature, pressure, and concentration of liquor as well as upon the duration of cooking, and not only different varieties but different growths of the same kind of straw give different results. Rape and leguminous straws are much more resistant to the action of alkali than grain straws, and sodium hydroxide gives better results than lime or sodium carbonate. By suitable treatment, a food material may be obtained which compares satisfactorily with hay or bran, grain straws giving the best results.

B. P. RIDGE.

Size of bamboo fibre and its variation with certain constituents. K. NISHIDA and K. WAKAMIYA (Cellulose Ind. Tokyo, 1927, **3**, 181—191).—The length of the

bamboo fibre increases as the stem is ascended to a maximum at about the 18th node and then decreases to the top, whilst the breadth of the fibre decreases continuously, though above the 18th node the change is small. Interior fibres are longer, broader, and contain less lignin than those in the exterior portion of the stem.

W. J. POWELL.

Behaviour of cellulose on heating. J. W. BAIN [with J. E. T. MUSGRAVE, G. F. KAY, G. M. CHUTE, and S. A. ROWLAND] (J.S.C.I., 1927, **46**, 193—195 T).—A study of the behaviour of cellulose when subjected to heat at temperatures well below that of exothermic decomposition. Cellulose (100 g.) heated at 210° for a long period underwent slow, progressive decomposition yielding, chiefly, carbon dioxide and monoxide, but after 34 days, when 2350 c.c. of gas had been obtained, the evolution became negligible and the residual dark brown brittle fibres were apparently stable towards heat. At 265° the evolution of gas (4100 c.c.) ceased after 376 hrs. Surgical cotton after extraction with hot water was heated for 3 hrs. at 200°, and the heated cotton extracted with 6 litres of boiling water; the straw-coloured extract on concentration yielded 0·2% of formic acid (calc. on the cotton), a small quantity of acetic acid, and 2·38% of a syrup which when freed from formic and other acids gave none of the usual colour reactions of pentoses or methylpentoses, but a fermentation test indicated the presence of a hexose. Phenylglucosazone and *p*-nitrophenylglucosazone were prepared from the syrup, whilst with *o*-nitrophenylhydrazine orange needles similar to lævulose-*o*-nitrophenylhydrazone were obtained. Mannose was not detected. The extracted cotton yielded further amounts of syrup on re-heating at 200°, the yield being almost as high as in the first extraction.

W. J. POWELL.

Value of p_H determination in the paper industry. R. ESCOURROU and P. CARPENTIER (Chim. et Ind., 1927, **18**, 13—23).—A scale of desirable p_H values for the different solutions used in the manufacture of sulphite pulp is given. The calcium bisulphite solution as it comes from tho towers has p_H about 2·0. After saturation with sulphur dioxide from the process the value is about 1·6. The proposed addition of salts such as chlorides or the use of sea water, which are stated to give better penetration of the wood, act only by modifying the p_H of the liquor. Chlorides increase hydrogen-ion concentration, sulphates diminish it. For any wood there is an optimum value. For resinous pinewood this is 2·1, which may be attained by addition of neutral sodium sulphite. The p_H value of the aqueous extracts of a number of kinds of wood is given. The variation of p_H during the process of cooking was systematically studied. It shows a rise to about 3·0 (period of impregnation), followed by a fall (dissolution of incrustants), and then by another rise (second impregnation). In the case of pulp for bleaching the last stage is omitted and the second is conducted slowly to as low a p_H value as possible, short of the point at which the liquor "turns." The measurement of p_H is, therefore, of great value in control. It is also of value in the washing of the pulp. In a particular case the fresh water used (p_H 8·1) was saturated with resins at p_H 6·1. A guide is thus furnished to the quantity of replacement water necessary, and to the possibility of the loss of soluble matter owing to faulty batches.

C. IRWIN.

Decomposition of vegetable matter under soils. TAYLOR.—See II.

Finnish "fluid resin." PYHÄLÄ.—See XIII.

Fermentation of pectin substances. OMELIANSKII and KONONOVA.—See XVIII.

PATENTS.

Increasing the electric insulating properties of cotton fibre and like insulating materials. LONDON ELECTRIC WIRE CO. & SMITHS, LTD., and E. L. WILDY (E.P. 272,587, 12.3.26).—The material, in the form of fibre, yarn, cloth, or tape, is impregnated under pressure first with a solution which precipitates protein (*e.g.*, 1% tannic acid), then with a solution containing protein (*e.g.*, 1% gelatin or glue), and finally dried.

B. P. RIDGE.

Production of artificial silk from cellulose or cellulose compounds. WOLFF & CO., E. CZAPEK, and R. WEINGAND (E.P. 257,925, 27.8.26. Conv., 1.9.25).—Rapid precipitation of aqueous cellulose solutions (viscose, cuprammonium, etc.) is obtained by the use of a bath consisting of a solution of an inorganic salt (magnesium chloride or sulphate) and/or an inorganic acid in methyl alcohol. Alternatively, a bath of pure methyl alcohol is first used, followed by the above. Filaments of extreme fineness can thus be produced owing to their high resistance to tearing.

B. P. RIDGE.

[Mechanically] cleaning plant fibres. F. KRUPP GRUSONWERK A.-G. (E.P. 262,761 and 274,716, 2. and 21.12.26. Conv., 12.12.25 and 6.8.26).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Dyeing of silks, viscose silk, and acetate silk. R. P. FREYSSINGEAS, R. E. E. WEILL, and M. H. DREGE (F.P. 606,487, 18.11.25).—A solution of 13·5 g. of cellulose octanitrate in a mixture of 67 c.c. of 95% alcohol and 67 c.c. of ether is added to a mixture of 54 c.c. of a vegetable oil, the dye, and 10 c.c. of paraffin oil, and diluted with alcohol-ether to the desired thinness.

C. HOLLINS.

Preparation of a printing paste for calico printing with cylinders. CHEM. FABR. VORM. SANDOZ (Swiss P. 115,536, 25.9.25).—To the printing colour is added an inert, highly viscous oil (mineral oil or tar oil), with or without paraffin wax etc. to increase the viscosity. These may be dissolved in hydrocarbons (heavy benzine oil), which minimise frothing. Acid printing pastes which contain basic dyes and starch thickening may further be mixed with high-molecular fatty acids (œnanthylic acid etc.) in addition to the viscous oils. C. HOLLINS.

Sizing for textiles. TEINTURERIE DE LA RIZE (F.P. 607,223, 1.12.25).—Air is blown through a mixture of castor oil, linseed oil, and manganese acetate or resinate at 100—105°. The yarn is impregnated with the product either in a vacuum or with addition of carbon tetrachloride. Oxidation on the fibre is prevented by rinsing with a dilute alcoholic solution of quinol or similar agent.

C. HOLLINS.

Treatment of vegetable fibrous materials with mercerising and like liquids. E. GMINDER (E.P.

262,154 and 267,470, [A] 30.11.26, [B] 14.12.26. Conv., [A] 30.11.25, [B] 13.3.26. [B] Addn. to [A]).—(A) Fabric saturated with the mercerising liquid is cooled in the stretching zone by being passed between pairs of rollers consisting alternately of stretching and cooling rollers. The material is subsequently fixed by washing in the stretched condition with hot water or dilute sodium hydroxide solution. (B) Instead of being washed with hot liquids in the stretching zone the stretched material is passed over heated rollers or surfaces, and then, if desired, again over cooled surfaces, the stretching in this case being partially or wholly dispensed with.

B. P. Ridge.

Machines for washing or otherwise treating fabrics with liquids. J. E. and A. Shaw (E.P. 274,703, 1.12.26).

Dyeing or like machines [for loose stock, rags, etc.]. I. Mitchell (E.P. 274,651, 31.7.26).

Dyes and dyeing (E.P. 274,156 and 274,303).—See IV.

Dyeing wood (E.P. 439,226).—See IX.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Influence of reaction rate on operating conditions in contact sulphuric acid manufacture. II. W. K. Lewis and E. D. Ries (Ind. Eng. Chem., 1927, **19**, 830—837).—A series of determinations of the rate of conversion of sulphur dioxide in a platinised asbestos converter was made under isothermal conditions and using dried gases. Owing to the exothermic character of the reaction such conditions can only be obtained with a low concentration of sulphur dioxide (up to 0·5 mol. %). This volume was diluted with either air, air and nitrogen, or air and sulphur trioxide, the latter being produced in a preliminary converter. With low concentrations of both sulphur dioxide and sulphur trioxide neither Knietsch's equation, based on the law of mass action, nor Bodenstein's equation was found to hold good, the results agreeing with the simple equation $dC/dt = K(SO_2)$. In other words, the effects of alterations in the concentration of oxygen or sulphur trioxide are negligible. This is a particular case of a general relationship of the form $-dx/dt = xf(r)$, where x is the number of sulphur dioxide molecules at time t and r is the ratio of sulphur trioxide to sulphur dioxide. The experimental results were in agreement with the relation $-dx/dt = bx(\log_e r_e - \log_e r)$, where r_e is the gas ratio at equilibrium. In this the only term influenced by the oxygen concentration is r_e which varies with $(O_2)^{0.5}$. This agrees with the small effect of oxygen variations. For given initial concentrations and given percentage conversion, there will be a temperature at which $-dx/dt$ will be a maximum. These have been calculated and agree with plant practice. The equation, unlike Bodenstein's, does not demand stoichiometric proportions of sulphur dioxide and oxygen since an increase in the former is accompanied by a decrease in r_e, and this also accords with general experience.

C. Irwin.

Analysis of mixed acid. Loriette and Jovinet (Mém. poudres, 1927, **22**, 174—179).—The usual method of analysing mixed acids can be shortened by determining the sum of nitrous and nitric acids by the Devarda method instead of by the nitrometer. By this modification it is possible to determine the sum on the same sample that was used in determining the total acidity. This application of the Devarda method has been made possible by the design of a special pipette, a description of which is given, which enables the mixed acid to be weighed out with the requisite accuracy. The weighed sample is run into water and the total acidity is determined, followed by a determination of the nitrogen in the neutralised liquid by the Devarda method. The values obtained agree well with those obtained by the nitrometer.

S. Binning.

Volumetric relationships in the burning of hydrogen sulphide to sulphurous acid. R. Nitzschmann and E. Vogel (Chem.-Ztg., 1927, **51**, 557—558).—The volumetric relationships in the equation $2H_2S + 3O_2 = 2SO_2 + 2H_2O + Q$ have been calculated. The results are expressed graphically.

L. M. Clark.

Removal and recovery of phenols from ammonium sulphate still effluents. D. W. Parkes (J.S.C.I., 1927, **46**, 186—193 T).—If the effluent from the distillation of ammoniacal liquor is saturated with carbon dioxide and allowed to settle, the phenols, present to the extent of 0·1—0·6%, may be removed by treatment with activated carbon. At first the phenols adsorbed decompose on the carbon, but this action ceases with time. The proportion adsorbed gradually falls, and thus the total phenols recovered, at first low, rise to a maximum and then fall off. Recovery is best effected with superheated steam and extraneous heating at 380—600°. Different types of activated carbon gave very varying results. Both coke and wood charcoal were used with fair success. The sample should be granulated, and should not only have a high saturation value, but should reach it quickly. Water is always adsorbed as well. The thiocyanate content of the liquor was found to show some reduction. The likelihood of success of this process depends on the securing of a suitable carbon. The best results obtained in laboratory experiments corresponded to a treatment rate of 2700 gals. per ton of carbon per hr., one ton of carbon being removed for separation of phenols every 4 hrs.

C. Irwin.

Conversion of alkali chlorides into nitrates with simultaneous production of chlorine. V. Dominik (Chim. et Ind., 1927, **18**, 24—32).—The aim of the process is to furnish cheap potassium nitrate for agricultural purposes. Krupp's "thermisilid-extra" (silicon-steel) is found to be practically unacted upon by aqua regia, and is available as constructional material. The reaction $HNO_3 + 3HCl = 2H_2O + Cl_2 + NOCl$ has been studied in detail. (It is reversed on dilution.) The proportions of alkali chloride, nitric acid, and mother-liquor necessary for a maximum evolution of chlorine are worked out. It is proposed to carry on the reaction in a vessel with a reflux condenser, and to separate the chlorine and nitrosyl chloride by liquefaction. The chlorine will be purified by washing with dilute nitric acid: $NOCl + Cl_2 + 2H_2O = HNO_3 + 3HCl$. Nitrosyl chloride will be decomposed by cold dilute nitric acid, $3NOCl + 2H_2O = HNO_3 + 3HCl + 2NO$, and the

nitric oxide oxidised. The 7—8N-solution of acids is returned to the process. For each kg. of sodium nitrate 0·54 kg. of water will be introduced into the process, and the direct yield of chlorine will be 60%. Allowing for water formed on the reaction and the use of 50% nitric acid, the total quantity of water to be eliminated will be 1·64 kg. per kg. of sodium nitrate. This will be performed in a concentration column, the liquor from which will be returned to the sodium nitrate ready for crystallisation. With sodium nitrate refrigeration will be necessary. With potassium nitrate the acid concentrate should not be added until after crystallisation.
C. Irwin.

Comparison of colorimetric methods for the determination of perchlorate. Junck and Küpper (Caliche, 1926, 8, 159—168).—The methods of Hofmann and Höbold (A., 1911, i, 608) and Hahn (B., 1926, 404) are discussed. For the colorimetric determination of perchlorate in crude nitrate a reagent is used consisting of 20 c.c. of zinc sulphate solution (d 1·35) and 5 c.c. of a solution of 1·6 g. of Methylene Blue in 5 litres of water; 5 c.c. of the reagent are shaken with 0·2 c.c. of 20% nitrate solution. After at least 1 hr. the colour is compared with those of ten standards prepared from similar nitrate solutions containing 0·1—1·0% of perchlorate. If more than 0·8% of perchlorate is present, a 10% nitrate solution is employed. The determination is accurate to 0·01%. Free iodine must be removed, but other usual impurities do not interfere. New standards are necessary in each new set of determinations.
Chemical Abstracts.

Chlorination of ilmenite. Y. F. Krieger (Contrib. Study Nat. Resources U.S.S.R. [Russia], [56], Titanium, 1926, **1**, 23—33).—A report of the titanium commission on the subjection of briquettes of ilmenite and coal to the action of chlorine not above 750°, and the redistillation and sublimation of the products.
Chemical Abstracts.

Chlorination of titanium carbide. M. S. Maksimenko and A. Eliseev (Contrib. Study Nat. Resources U.S.S.R. [Russia], [56], Titanium, 1926, **1**, 43—47).—The apparatus described requires only 750—1000 watts per 8—9 kg., does not clog, and gives only small quantities of ferric chloride.
Chemical Abstracts.

Production of titanium carbide. M. S. Maksimenko (Contrib. Study Nat. Resources U.S.S.R. [Russia], [56], Titanium, 1926, **1**, 33—43).—After removal of dolomite, ilmenite is fused electrically with carbon; the slag is ground and magnetically separated, whereby the iron content is reduced from 34 to 12%, and the carbon content increased to 1·5%. Slag, coal, sawdust, and sodium chloride are then heated in an electric furnace; the iron content falls to 5% and the carbon content rises to 18%. Calcium carbide, magnesium chloride, and iron are removed by means of hydrochloric acid.
Chemical Abstracts.

Production of titanium dioxide, carbide, and tetrachloride. V. S. Suirokomskii (Contrib. Study Nat. Resources U.S.S.R. [Russia], [56], Titanium, 1926, **1**, 49—53, 53—55, 47—49).—A review.
Chemical Abstracts.

Determination of iodine in mother-liquor and in wash-liquors. C. C. Smith (Caliche, 1926, 8, 149—150).—To the liquor (10 c.c.) are added water (40 c.c.), 20% sulphuric acid (5 c.c.), and 0·1N-sodium hypochlorite in slight excess; the mixture is boiled until it occupies 35—40 c.c., cooled, diluted with water (300 c.c.), mixed with an excess of potassium iodide, and titrated with 0·1N-sodium thiosulphate. Chromates, if present, must be eliminated or separately determined.
Chemical Abstracts.

Determination of very small amounts of yellow phosphorus in red phosphorus. R. H. Kray (Ind. Eng. Chem., 1927, **19**, 816).—The colour produced on a copper sulphate test paper by a filtered solution of the red phosphorus in carbon disulphide is matched against those produced by known amounts of yellow phosphorus.

Rate of growth of crystals. Montillon and Badger. —See I.

Silica gel. Mautner.—See I.

Adhesives and adhesion. McBain and Lee.— See I.

Explosive ammonium salts. Kast.—See XXII.

Hydrogen peroxide explosives. Bamberger and Nussbaum.—See XXII.

Patents.

Direct production of acetic acid from crude pyroligneous acid. E. D. Deperrois (F.P. 601,156, 18.10.24).—The crude acid vapours from which the tar has been removed, and at a temperature at which acetic acid is just volatile, are sent through an apparatus in which the acid is condensed in a more or less diluted form in various chambers by suitable arrangement of heating or cooling coils, whilst the lowest-boiling fractions are not condensed and escape. The apparatus can be used for other similar separations. A. B. Manning.

Manufacture of solid sodium hypochlorite preparations. M. P. Appleby and C. Carter (E.P. [a] 274,197 and [b] 274,196, 16.4.26).—(a) A strong solution or melted crystals of sodium hypochlorite is mixed with anhydrous or partially dehydrated salts of a definite alkaline character capable of forming stable hydrates, *e.g.*, sodium metaborate or tripotassium phosphate. (b) The hypochlorite is admixed with dehydrated trisodium phosphate or its salts, or dehydrated disodium hydrogen phosphate and free caustic soda.
H. Royal-Dawson.

Manufacture of ammonium chloride. J. W. Moore, W. G. Polack, and Castner-Kellner Alkali Co., Ltd. (E.P. 274,263, 25.6.26).—Interaction is effected between synthetic ammonia and a gaseous mixture of hydrogen and hydrogen chloride (obtained by burning chlorine in an excess of free hydrogen) in a reaction chamber, the residual hydrogen being again used in the cyclic process.
H. Royal-Dawson.

Removal of silica from zinc, copper, and vanadium solutions. R. H. Stevens, G. C. Norris, and W. N. Watson (E.P. 274,297, 23.8.26).—The solution from the roasted ore is agitated and heated at 40—65°, in the presence of a basic material, *e.g.*, zinc oxide, zinc dross, lime, producing a granular precipitate of silica, which is filtered off from the resulting pulp, the filter cake being then washed with weak acid and water till free of its metal contents.
H. Royal-Dawson.

Continuous production of pure carbon disulphide, sulphur, and highly concentrated hydrogen sulphide from crude carbon disulphide. I. G. FARBENIND. A.-G., Assees. of P. SIEDLER and E. SCHULTE (G.P. 439,766, 25.10.25).—Two similar fractionating columns, *A* and *B*, attached to reflux condensers are employed. *A* is maintained above 120° and is fed with crude carbon disulphide from above; down *B*, which is kept just above 47°, the purified carbon disulphide from *A* is allowed to trickle. The vapours may be withdrawn from the lower end of the columns and raised to the level of the upper end before complete condensation. The gases from the exits of the columns may be condensed and returned to column *A*. C. HOLLINS.

VIII.—GLASS; CERAMICS.

Changes in optical glasses. P. NICOLARDOT (Rev. gén. Colloid., 1927, **5**, 539—543).—An account is given of the methods used by different investigators for measuring the degree of resistance of glasses to various factors (such as the action of atmospheric moisture or carbon dioxide, the presence of hydrocarbon vapours from lubricants, etc.) causing superficial changes in the glass. The method of Mylius (Ann. Phys. Chem., 1879, **6**, 431) is described in detail; this is based on the determination of the quantity of alkali yielded by the glass, using erythrosin as indicator.

L. L. BIRCUMSHAW.

Behaviour of fluorides in glazes and enamels. G. AGDE and H. F. KRAUSE (Z. angew. Chem., 1927, **40**, 886—895).—Examination of the waste gases from a furnace melting fluoride enamels revealed no formation of silicon fluoride. From the analysis of a series of glazes previously studied for opalescence (B., 1927, 483) fluorine losses of 12—34% were found, the chief cause of which was the volatilisation of sodium and aluminium fluorides, whilst the formation of some hydrofluoric acid was also indicated. A. COUSEN.

Use of super-refractories as chequer brickwork in [oil-]gas manufacture. H. J. KNOLLMAN (J. Amer. Ceram. Soc., 1927, **10**, 299—308).—With the object of increasing gas-generating capacity, carbofrax and diamel (fused magnesia spinel) bricks, possessing superior heat-transfer properties, were tried in the upper chequer brick section of a full-sized, standard, single-shell, oil–gas generator. Although the object was not realised, an analysis of the reasons for this suggests excellent possibilities for such products in oil–gas production. A. T. GREEN.

Effect of steam on the transverse strength of fireclay bricks. C. W. PARMELEE and A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1927, **10**, 292—298).—Transverse strength data on twelve of each of three brands of fireclay bricks, the composition and properties of which are detailed, and which had been subjected to the action of steam at 1100° for 10 hrs. and to an ordinary firing at 1100° for the same period, were obtained and compared with similar data obtained on untreated samples of the same products. The results indicate that steam has no influence on the strength of the firebricks investigated. The colour of the steamed and of the reheated samples was much darker than that of the new bricks, the "iron" spots showing up in a much more pronounced manner. Further tests with more varieties of fireclay refractories are suggested.

A. T. GREEN.

PATENTS.

Protecting the silvering of glass. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY, & CIREY (E.P. 262,824, 13.12.26. Conv., 14.12.25).—A stable and uniform backing for silver layers on glass is prepared from a synthetic resin, such as a phenol–formaldehyde condensation product, of which 40 pts. are dissolved in about 60 pts. of a solvent such as denatured alcohol. The liquid is spread as a thin film on the dried silver coating and the glass gradually heated to, and kept at, about 93° for 2 hrs.

A. COUSEN.

Refractory, acid-proof and other ceramically bonded products. SCHEIDHAUER & GIESSING A.-G. (E.P. 263,194, 18.12.26. Conv., 19.12.25).—Firebricks, blocks, etc. are made from a mixture of kaolin, liquefied by water and an electrolyte as deflocculator, together with a finely ground non-plastic material such as is usually used for refractories or ceramically bonded articles, the mixture being in such proportions that it can be beaten, stamped, or pressed to shape. After drying, the articles may be burned *in situ*. Mixing may be rapidly effected by first liquefying the kaolin, together with part of the non-plastic material, then adding the remainder. For masses of low plasticity more water than necessary for making the slip is preferably added, the excess being removed subsequently if necessary for moulding purposes. A. COUSEN.

IX.—BUILDING MATERIALS.

Temperature developed in high-alumina concrete. T. H. CUTLER (Eng. News-Rec., 1927, **99**, 146).—High-alumina concrete laid under cold weather conditions began to increase in temperature about 4 hrs. after mixing and generated enough heat during the hardening period to prevent its freezing, without protection, when air temperatures fell slightly below 0°. Maximum temperatures of 29° and 23°, respectively, for the interior and the surface of the concrete were attained in 9 to 10 hrs. W. T. LOCKETT.

Penetrance of oily fluids in wood. A. M. HOWALD (Chem. Met. Eng., 1927, **34**, 413—415).—Petroleum residues (fuel oil), alone or mixed with creosote, in some cases have a much lower penetrance in wood than their viscosities would indicate, which is not the case with petroleum distillates. A similar reduction in the penetrance of a distillate was produced by adding magnesium oleate or stearine pitch. These form colloidal solutions in oil, and it is suggested that the reduction in penetrance caused by the addition of asphaltic residues is similarly caused. The addition of carbon black in each case increased the penetrance, presumably by removing the peptised colloid. C. IRWIN.

PATENTS.

Manufacture of cement. SOC. ANON. DES CHAUX ET CIMENTS DE LAFARGE ET DU TEIL (E.P. 250,246, 31.3.26. Conv., 4.4.25).—Aluminous cement is prepared by heating an intimate mixture of ferruginous bauxite

and lime or limestone, each ground so that at least 90% passes the 180-mesh per inch sieve, at a temperature below sintering (*i.e.*, 1000—1100°) for about 6 hrs. The mixture may be such as to give the constituents within the approximate limits, silica 0—5%, iron oxide 10—25%, alumina 40—55%, lime 20—40%.

A. Cousen.

Process of manufacturing acid-proof hydraulic binding agents. Portland-Cementwerk Balingen G.m.b.H. (E.P. 269,549, 12.4.27. Conv., 13.4.26).—Barium sulphate or witherite, mixed with sand, clay, or other materials rich in silicic acid, is burnt to the sintering or fusing point, preferably with an admixture of coal or coke. The process is arranged so that the percentage of barium aluminate is kept low, and a proportion of aluminium silicate or iron oxide is formed. The ground product is highly resistant to acids, sulphates, etc.; the resistance is increased by treatment with a sulphate solution. B. W. Clarke.

Manufacture of artificial stone. R. H. Abrey (E.P. 273,989, 14.2.27).—Raw or calcined magnesite is heated with a flux, *e.g.*, sodium silicate or borax, to the sintering point, and the ground product is gauged with a solution of magnesium chloride containing aluminium chloride and barium chloride. B. W. Clarke.

Through dyeing of wood. E. Ambühl (G.P. 439,226, 4.2.25. Conv., 26.9.24).—The wood, sawn into planks, is introduced, moist or oven-dried, into an autoclave containing the colouring solution, and a pressure of 30—40 atm. is applied. The heart-wood and annual rings remain uncoloured. After about 5 hrs. the pressure is released to 5 atm. and the liquid is boiled to soften the wood, after which the pressure is again increased to 20, 30, or 40 atm. for 2 hrs. The contents are then allowed to cool at this pressure for 6—12 hrs. and the pressure is finally released slowly. The wood, now coloured throughout, is piled with good access of air, and after some days is dried in a drying plant. Water-soluble aniline dyes or colours soluble in alcohol or glycerol are employed and give an odourless wood. Greater depths of shade are obtained by evaporation, lighter shades by merely boiling. C. Hollins.

[Bituminous] material for road surfaces. J. F. Bennett and J. Hadfield (E.P. 274,248, 4.6.26).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Deformations accompanying the thermal treatment of steel. A. Portevin and A. Sourdillon (Rev. Mét., 1927, **24**, 215—233).—The test-pieces examined diminished in length and increased in diameter after each anneal, the cross-section tending to become ellipsoidal. The effect of successive anneals was additive and more marked the higher the annealing temperature, but was independent of time. The variations in dimensions increased greatly with the rapidity of cooling above 600°; below this temperature the rate of cooling had no effect. In quenching experiments the deformations depended on the quenching temperature, and might be positive or negative; *e.g.*, a semi-hard steel quenched from 850° showed an increase in length and a decrease in diameter. For each steel there exists a certain quenching temperature such that no variations in length are produced, and another temperature at which there are no variations in diameter. With extra-hard carbon steels oil-quenching produced less deformation than water-quenching. The duration of heating before quenching had a very slight influence, whilst one anneal annuls, from the point of view of the deformations, the effects of all previous heat-treatments.

M. E. Nottage.

Reflecting power of some unoxidisable steels. (Mlle.) J. Clavier (Compt. rend., 1927, **185**, 264—266).—Certain so-called "unoxidisable" steels have excellent reflecting powers which are not impaired on exposure to air. They may also be silvered without being attacked.

J. Grant.

Variations in the mechanical properties of steels and alloys with temperature. A. Michel and M. Matte (Rev. Mét., 1927, **24**, 200—209).—A wire under a constant load and maintained at a constant temperature above that of its surroundings for a sufficiently long period of time undergoes several kinds of elongations, viz.:—(1) an instantaneous elastic elongation, (2) a slow elongation which proceeds with decreasing rapidity, and can be resolved into an elastic elongation with retarded elasticity or reactivity, and a permanent elongation, (3) if the load is sufficient, the foregoing elongation is followed by another which proceeds with constant rapidity and corresponds to a so-called "viscous" deformation, (4) after a certain time the rapidity of elongation increases very rapidly up to the breaking-point of the test-piece; this period corresponds to the striction. Each of these quantities should be evaluated separately when studying the behaviour of metals and alloys under mechanical stretching forces at different temperatures. The influence of the time factor in tests made at high temperatures is of the greatest importance. To determine the viscosity limit a study of the variations of the elastic limit and of the modulus of elasticity as a function of the temperature is necessary. M. E. Nottage.

Determination of silicon in ferrosilicon. L. Deutsch (Chem.-Ztg., 1927, **51**, 558—559).—Ferrosilicon is best decomposed by fusion with sodium hydroxide and potassium nitrate. The fused mass is acidified with hydrochloric acid and the silicon determined as silica after treatment by the usual methods. L. M. Clark.

Nickel and nickel–chromium in cast iron. A. B. Everest (Bull. Brit. Cast Iron Res. Assoc., 1927, **16**, 14—19).—Nickel increases the quantity of graphitic carbon similarly to silicon and aluminium. It improves machinability and resistance to wear. Grain growth on heating is about 15% of that of foundry iron. The effect is believed to depend on the silicon and phosphorus content. Graphitisation may be retarded by addition of chromium (0·5%); the iron is then hard and close-grained, but readily machinable.

Chemical Abstracts.

Chromium plating and resistance to corrosion. S. Wernick (Nature, 1927, **120**, 225).—The limitations of chromium as a corrosion preventive are briefly mentioned. A. A. Eldridge.

[Chromium plating and resistance to corrosion.]

H. C. H. C. (Nature, 1927, **120**, 225—226).—A reply to Wernick (preceding abstract). A. A. Eldridge.

Titanium and its application in the industries. V. S. Suirokomskii (Contrib. Study Nat. Resources U.S.S.R. [Russia], [56], Titanium, 1926, **1**, 76—108).—A comprehensive review. Chemical Abstracts.

Analysis of Babbitt metal. I. B. Mintz, B. O. Lyubin, and V. I. Zilberman (Nauch. Zapiski [Russia], 1927, **4**, 196—198).—The filings (1 g.) are evaporated to dryness with water (5 c.c.) and concentrated nitric acid (10 c.c.), copper and lead salts being then extracted from the residue with $7 \cdot 5N$-nitric acid. The residue is washed with ammonium nitrate solution, and the filtrate, after addition of 10 c.c. of concentrated nitric acid, is electrolysed for copper and lead. To determine antimony and tin, 0·5—1 g. of the metal is dissolved in 10 c.c. of concentrated sulphuric acid, boiled for 5—7 min., cooled, mixed with water (50 c.c.) and concentrated hydrochloric acid (20 c.c.), boiled for 2 min., quickly cooled, diluted with water (100 c.c.), and the antimony titrated with $0 \cdot 1N$-permanganate. The solution is poured into $6N$-hydrochloric acid (110 c.c.), iron wire (1·5 g.) added, heated slowly in a current of carbon dioxide, and, after dissolution of all the iron, cooled, the tin being titrated with $0 \cdot 1N$-iodine solution. Chemical Abstracts.

Separation of chromium, tungsten, molybdenum, and vanadium and application of the method to the analysis of stellite alloys. E. Cremer and B. Fetkenheuer (Wiss. Veröff. Siemens-Konz., 1927, **5**, 199—203).—The substance is fused with 10 g. of a mixture of 12 pts. of sodium carbonate, 7 pts. of potassium carbonate, and 1 pt. of potassium nitrate in a platinum crucible. The product is extracted with water, and the filtered solution boiled with an excess of hydrochloric acid, which precipitates the greater part of the tungstic acid. The remainder of the tungsten is recovered from the filtrate by the usual evaporation to dryness, followed by heating at 120° and extracting the residue with dilute hydrochloric acid. The combined tungstic acid precipitates are weighed, and fused with sodium carbonate, and the solution of the mass is treated with tartaric acid, and saturated with hydrogen sulphide under pressure to recover the trace of molybdenum precipitated with the tungsten. The main portion of the molybdenum is recovered from the hydrochloric acid solution by means of hydrogen sulphide under pressure, and both precipitates are ignited to oxide for weighing. The filtrate from the hydrogen sulphide treatment is boiled to remove excess of the gas, and treated with the minimum amount of sodium hydroxide to precipitate chromic hydroxide, which is collected, washed, ignited, and fused with sodium carbonate in a current of coal gas, whereby the occluded vanadium dissolves as sodium vanadate. The aqueous extract of the fused mass is added to the main vanadium filtrate, which is just acidified with hydrochloric acid and treated in succession with manganous chloride, hydrogen peroxide, and ammonia. The precipitate of manganous vanadate and peroxide is dissolved in hydrochloric acid, the solution evaporated with sulphuric acid, and the vanadium determined volumetrically in the usual way. The chromic oxide residue is fused with sodium peroxide for volumetric determination of the chromium. In applying this method to the analysis of stellite alloys the finely-pulverised alloy may be dissolved by the fusion mixture described above, or better by fusion with potassium hydrogen sulphate. In the latter case the solution of the melt in dilute sulphuric acid is treated with 1 c.c. of bromine, followed by sufficient 5% sodium hydroxide solution to precipitate all the iron, manganese, cobalt, and nickel. The precipitate is collected, washed, dissolved in hydrogen peroxide and sulphuric acid, and the precipitation with bromine and sodium hydroxide is repeated. The filtrates from both precipitations are united, treated with hydrochloric acid and ammonia to separate the silica and alumina, and the analysis is finished as described above. A. R. Powell.

Practical application of inhibitors in [metal] pickling operations. F. N. Speller and E. L. Chappell (Chem. Met. Eng., 1927, **34**, 421—423).—Inhibitors are commonly added to the acid baths in which steel is pickled in order to prevent dissolution of the metal, while allowing that of oxide to continue. Amongst inhibitors used have been nitrogen bases derived from coal-tar, mouldy flour or bran, glue, and vitriol tar sludge from benzol washing. The efficiency of certain of these was determined by measurement of the rate of evolution of hydrogen. To give results of practical value this must be done at the temperature at which treatment is to be actually applied. In general, increase of concentration of inhibitor gives a diminishing effect, so that for a given inhibitor there is a concentration which it does not pay to exceed. Amongst the advantages gained are saving in acid, preservation of metal surface, avoidance of "hydrogen embrittlement," and a reduction in acid fumes. C. Irwin.

Dispersoid chemistry in metallurgy. F. Sauerwald (Kolloid-Z., 1927, **42**, 242—253).—A number of subjects are dealt with, including the influence of the state of division on the mechanical properties of solid metals; the differences between the mechanical properties of single crystals and crystal aggregates; general views on the relation between inter- and inner-crystalline cohesions, with special reference to Rosenhain's work on the mode of fracture of metals; the influence of cold rolling (cf. A., 1927, 302); and the connexion between the degree of dispersion and the separation of mixed crystals. L. L. Bircumshaw.

Corrosive effect of sulphur etc. in naphtha solution. Schmidt.—See II.

Patents.

Blast furnace. J. A. Parker (U.S.P. 1,629,045, 17.5.27. Appl., 7.7.25).—The lower edge of the furnace wall comprises a series of blocks separated by water spaces. The blocks extend the entire width of the wall, and their lower faces are alined with the lower faces of the water spaces. Air passages extend through each block into the furnace. H. Holmes.

Age-hardening aluminium-silver alloys. W. Kroll (Metall u. Erz, 1926, **23**, 555—557; Chem. Zentr., 1927, I, 175; cf. B., 1926, 751).—The solubility of silver in solid aluminium rises from 1·3% at 20° to about 9%

at 525°, consequently silver–aluminium alloys within this range exhibit the phenomenon of age-hardening when quenched from above 525° and subsequently aged at 130°. The maximum tensile strength so obtained is less than that of the corresponding copper–aluminium alloys by about 5—8 kg./mm.^{2} A. R. Powell.

Alloy [for hard-chilled castings]. J. N. Early (U.S.P. 1,622,576, 29.3.27. Appl., 21.7.26).—A ferrous metal alloy, suitable for the making of castings such as rolling-mill rolls and capable of forming a controllable chill in a sand mould without the use of chills, contains 0·75—1·50% Si, 0·05—0·35% S, 0·25—0·75% P, 0·20—0·70% Mn, 2·00—3·75% C, 0·05—0·25% Ni, 0·15—0·25% Cr, 0·10—0·30% Mo, and remainder iron. M. E. Nottage.

Plating metals [iron]. B. S. Summers, Assr. to Locomotive Terminal Improvement Co. (U.S.P. 1,619,835, 8.3.27. Appl., 9.2.24).—An alloy of lead and nickel is deposited electrolytically on iron from a bath containing lead acetate, sodium hydroxide, nickel chloride, potassium cyanide, and glue. A dense, resistant coating is obtained. T. S. Wheeler.

Aluminium alloy containing lithium. J. Czochralski, Assr. to Allied Process Corp. (U.S.P. 1,620,082, 8.3.27. Appl., 7.12.23).—A hard alloy of good tensile strength comprises Li up to 0·5%, Zn 1—12%, Cu up to 3%, and aluminium. The properties are improved by heating at 100—500° and cooling. T. S. Wheeler.

Smelting of antimony, arsenic, and mercury ores. Oesterr. Bamag-Büttner-Werke A.-G., and R. Jahn (Austr. P. 104,390, 7.8.25. Addn. to Austr. P. 102,293; B., 1926, 549).—To economise in fuel the ore is heated nearly to the desired roasting temperature during the drying operation, and, when quite dry, subjected to a roasting or chlorination treatment, the resulting oxide or chloride being subsequently reduced as described in the chief patent. A. R. Powell.

[Gold] alloy. Western Electric Norsk A./S. (Dan. P. 34,635, 29.7.24).—A gold alloy suitable for electrical contacts contains 72% Au, 26·2% Ag, and 1·8% Ni. A. R. Powell.

[Platinum-copper] alloy. C. S. Brainin, Assr. to Baker & Co., Inc. (U.S.P. 1,624,857, 12.4.27. Appl., 17.10.22).—An alloy of high electrical resistance contains at least 75% Pt and the remainder copper. F. G. Crosse.

Electrolytic process and apparatus [electrodeposition of metals]. H. C. Harrison (E.P. 274,405, 5.1.26).—Electrolyte flows through the electrolytic bath in two streams, the one flowing with a high velocity through a tunnel-like cathode compartment in contact with the cathode, which is moved through the compartment, and the other stream flowing at a lower speed through a compartment containing the metal to be deposited, *e.g.*, copper, the two streams subsequently mixing. J. S. G. Thomas.

Apparatus for concentrating ores etc. by flotation. F. P. Egeberg, Assee. of J. C. MacIntosh (E.P. 261,395, 11.11.26. Conv., 13.11.25).—See U.S.P. 1,608,896; B., 1927, 115.

[Annealing and like] furnaces. A. Smallwood and J. Fallon (E.P. 274,730, 22.1.27).

Manufacture of converter bottoms. J. Schmidt (E.P. 274,316, 11.10.26).

Electroplating [apparatus]. H. Göthe (E.P. 274,671, 14.9.26).

XI.—ELECTROTECHNICS.

Patents.

Pasted accumulator grid or plate. W. Haddon and J. M. Burnett (E.P. 274,208, 19.4.26).—The pasted plate is coated with a mixture of rubber latex and a salt soluble in water, *e.g.*, magnesium sulphate or ammonium carbonate, then dried, and the soluble salt washed out. J. S. G. Thomas.

Manufacture of oxide cathodes. H. Wade. From N.V. Philips' Gloeilampenfabr. (E.P. 274,233, 30.4.26).—A core of highly refractory metal or alloy, *e.g.*, tungsten or molybdenum, which does not form an alloy with the alkaline-earth metals is coated with a film of readily oxidised metal, *e.g.*, copper, and the film then oxidised and brought into contact with one or more of the alkaline-earth metals, which are finally converted into oxide, and the whole is heated to evaporate any remaining reduced readily oxidised metal contained in the film. J. S. G. Thomas.

Induction furnaces. British Thomson-Houston Co., Ltd., Assees. of M. Unger (E.P. 263,856, 30.12.26. Conv., 31.12.25).—See U.S.P. 1,626,437; B., 1927, 450.

Treating filaments for electric incandescence lamps etc. [by spraying]. British Thomson-Houston Co., Ltd., Assees. of G. E. Inman (E.P. 270,733, 5.5.27. Conv., 10.5.26).

Treating carbonaceous material (E.P. 267,240—1).—See II.

Increasing insulating properties of cotton etc. (E.P. 272,587).—See V.

Electrodeposition of metals (E.P. 274,405).—See X.

XII.—FATS; OILS; WAXES.

Chemistry of the [fat of] edible domestic birds. J. S. Hepburn and A. B. Katz (J. Franklin Inst., 1927, **203**, 835—841).—The constants of the visceral fat of the turkey (*Meleagris gallopavo*) and the goose (*Anser anser*), the subcutaneous fat of the goose, and the egg fat of the duck (*Anas erythrorhynchos*) and the goose were determined. The following results were recorded for goose and duck eggs, respectively:—Average weight, 133·0, 80·3 g., the shell being 12·72, 10·58%, and the edible portion 87·28, 89·42% of the gross weight. The yolk formed 36·86, 38·08%, and the white 63·14, 61·92% by wt. of the edible portion. The gross composition (%) of the edible portion of duck and goose eggs, respectively, is tabulated for (*a*) whole egg, (*b*) white, and (*c*) yolk. Thus, for total solids, (*a*) 31·45, 38·75; (*b*) 12·98, 12·08; (*c*) 56·31, 58·90; ether extract, (*a*) 14·36, 12·20; (*b*) 0·01, 0·06; (*c*) 35·04, 35·74; crude protein, (*a*) 13·64, 14·12; (*b*) 9·88, 10·77; (*c*) 18·42, 18·67. The percentage composition of the abdominal adipose connective tissue of the turkey and goose respectively is:—Total solids, 92·94, 91·46;

moisture, 7·06, 8·54; ether extract, 90·2, 88·12; crude protein, 2·24, 2·57. The following constants are given of the visceral fat of the turkey; the visceral, subcutaneous, and egg-yolk fats of the goose; and the egg-yolk fat of the duck, the respective values being:—Acid value, 6·8, 3·4, 0·5, 4·7, 4·8; saponif. value, 225·1, 211·6, 216·5, 199·2, 205·1; ester value, 218·3, 208·2, 216·0, 194·5, 200·3; Hehner number, 90·87, 89·58, 94·90, 87·10, 87·46; iodine number, Hübl, 65·5, 68·6, 73·4, 63·3, 70·8; soluble acids, 5·48, 4·16, nil, 2·57, 2·66; n^{20}, 1·4663, 1·4647, 1·4659, 1·4651, 1·4674; phosphorus (%), 0·074, 0·165, 0·156, 0·927, 0·628. H. M. LANGTON.

Marine animal oils. Aliphatic unsaturated alcohols in spermaceti oil. E. ANDRÉ and (Mlle.) M. T. FRANÇOIS (Compt. rend., 1927, **185**, 279—281; cf. Toyama, A., 1924, i, 257).—The separation of oleyl alcohol (Bouveault and Blanc, A., 1905, i, 12) from spermaceti oil by Tsujimoto (Chem. Umschau, 1921, **28**, 71) has been repeated. The liquid alcohols in the unsaponifiable matter were converted into the mixed acetates, which were fractionally distilled. After some hexadecyl acetate, the main fraction was *oleyl acetate*, b.p. 200—203°/3 mm., which had iodine and saponification values very near the theoretical. In the higher fractions an *acetate* was found which gave a *tetrabromide*, $C_{22}H_{42}OBr_4$, m.p. 242°. Oleyl phenylurethane had not a sharp m.p. (cf. Bouveault and Blanc, *loc. cit.*), but the β-*naphthylurethane* had m.p. 44—45°. *Oleyl allophanate* was not obtained homogeneous; of two portions, m.p. 135° and 129°, the former saponified to a mixture of oleyl and elaidyl alcohols. Elaidyl alcohol could not easily be separated from the product of the action of nitrous fumes upon oleyl alcohol; prepared by the method of Toyama from elaidic acid (with which it shares the property of an iodine value below the theoretical), it had m.p. 36—37°, *phenylurethane* m.p. 55—56°, β-*naphthylurethane* m.p. 71°. Elaidyl alcohol is more stable than oleyl alcohol, which is apt to isomerise.
E. W. WIGNALL.

Fatty acids of shark and ray liver oils. V. Fatty acids of Aburatsunozame. Y. TOYAMA and T. TSUCHIYA (J. Soc. Chem. Ind., Japan, 1927, **30**, 207—215).—The oil of Aburatsunozame (*Squalus wakiyae*, Tanaka) contains a considerable amount of unsaponif. matter (16·79% of the oil), which consists essentially of alcohols of the selachyl alcohol group (cf. Toyama; B., 1924, 431, 719; 1927, 82, 83). The fatty acids of the oil contain more than 15% of saturated acids. The unsaturated acids contain a considerable amount of acids more unsaturated than those of the oleic series. The saturated acids consist mainly of palmitic acid with small quantities of myristic, stearic, arachidic, and behenic acids, and an acid $C_{24}H_{48}O_2$. The acids of the oleic series consist of zoömaric and oleic acids, an acid $C_{20}H_{38}O_2$, and cetoleic and selacholeic acids, possibly with an extremely minute amount of an acid $C_{14}H_{26}O_2$. The acids more unsaturated than those of the oleic series consist chiefly of C_{22} and C_{20} acids, and possibly also of a small amount of C_{18} acids. Among the C_{18} acids, $C_{18}H_{28}O_2$ and $C_{18}H_{30}O_2$ seem to be present. $C_{20}H_{32}O_2$ seems to form the main constituent of C_{20} acids. C_{22} acids consist mainly of $C_{22}H_{34}O_2$. K. KASHIMA.

Oils and fats from the seeds of Indian forest plants. VIII. Oil from the seeds of *Thevetia Neriifolia* (Juss.). R. BHATTACHARYA and P. R. AYYAR. **IX—XI. Oils from the seeds of *Cerbera odollam*, *Holarrhena antidysenterica*, and *Anona squamosa* (Linn.).** R. V. GHANEKAR and P. R. AYYAR (J. Indian Inst. Sci., 1927, **10A**, 15—31).—VIII.—Extraction of the decorticated seeds of *Thevetia Neriifolia* yields 57% of a pale yellow oil (d_{15}^{15} 0·903, n_D^{40} 1·4599, acid value 4·3, saponif. value 19·41, unsaponifiable matter 1·4%, acetyl value nil, iodine value 76·0, Polenske value 0·5, Reichert–Polenske value 0·4, Hehner value 95·6) consisting of the glycerides of palmitic (17·1%), stearic (11·8%), arachidic (0·47%), oleic (64·3%), and linoleic (6·3%) acids. The mixed fatty acids have n_D^{40} 1·4531, iodine value 77, solidif. pt. 34°, and mol. wt. 277·5. The unsaponifiable matter contains 15·3% of sitosterol.

IX.—Extraction of the seeds of *Cerberaodollam* (Gaertn.) yields 43·1% of a pale yellow oil (d_{15}^{15} 0·9144, n_D^{60} 1·4578, acid value 0·35, saponif. value 19·1, unsaponifiable matter 0·8%, acetyl value nil, iodine value 73·8, Polenske and Reichert–Polenske values inappreciable, Hehner value 94·8) containing the glycerides of linoleic (16·4%), oleic (4·2%), myristic ? (0·4%), palmitic (30%), stearic (9·9%), and lignoceric (0·9%) acids. The mixed fatty acids have n_D^{60} 1·4478, iodine value 75·8, solidif. pt. 33·8°, mol. wt. 277·9. The unsaponifiable matter contains 32·2% of a sterol, probably sitosterol.

X.—Extraction of the ground seeds of *Holarrhena antidysenterica* yields 19% of oil (d_{15}^{15} 0·9354, n_D^{60} 1·4666, acid value 36·1, saponif. value 180·5, unsaponifiable matter 3·5%, acetyl value 22·9, iodine value 149·1, Reichert–Meissl value 1·7, Reichert–Polenske value 0·4, Hehner value 94·3) composed of the glycerides of linolenic (10%), linoleic (54·7%), oleic (21%), palmitic (5·6%), stearic (6·8%), and lignoceric (1·9%) acids. The mixed fatty acids have n_D^{60} 1·4597, iodine value 151·3, solidif. pt. 24·7°, mol. wt. 283·8. The unsaponifiable matter contains 17·4% of phytosterol.

XI.—Extraction of the seeds of *Anona squamosa* (Linn.) dried at 110° (loss in wt. 33·3%) yields 30% of oil (d_{15}^{15} 0·9126, n_D^{60} 1·4558, acid value 0·8, saponif. value 188·3, unsaponifiable matter 0·2%, acetyl value 18·5, iodine value 85·6, Polenske value 0·1, Reichert–Polenske value 0·6, Hehner value 93·8) composed of the glycerides of oleic (18·1%), linoleic (55·2), palmitic (14·7%), stearic (10·7%), and cerotic ? (0·9%) acids. The mixed fatty acids have n_D^{60} 1·4470, iodine value 84·8, solidif. pt. 31·8°, mol. wt. 276·3, and contain an unidentified saturated liquid acid (equivalent 126·5). The unsaponifiable matter contains 15% of sitosterol.
E. H. SHARPLES.

Oil from seeds of *Bombax heptaphyllum*. A. C. DA S. TELLES (Bol. Min. Agr. Ind. Com., 1927, **16**, 178—181).—The oil from the seeds of *Bombax heptaphyllum*, Kunth, has *d* 0·920 and m.p. —9°. It is a drying oil. G. W. ROBINSON.

Grape stone oil. L. MARGAILLAN (Compt. rend., 1927, **185**, 306—307).—Oil carefully extracted from fresh grape stones has an acetyl value which may be as low as 4; it is thus, contrary to accepted opinion, unoxidised when extracted. On keeping, ground grape

stones become heated, owing to oxidation of the oil. Oxidation by "blowing" is slower than that of colza oil; the product, a jelly, is a good lubricant.

E. W. Wignall.

Origin of the coloration of beeswax. Composition of propolis. G. F. Jaubert (Compt. rend., 1927, **184**, 1134—1136).—The matter exuded by the wax glands of the bee is a colourless, transparent liquid, and on cooling forms translucent, pearly white scales. Extracted beeswax is, however, of a bright yellow colour, which is derived from the propolis varnish which covers the tops of the cells. The colouring matter of propolis combines with certain salts, such as those of iron, aluminium, copper, titanium, uranium, etc., to form insoluble lakes. From these the pure colouring matter may be isolated and is found to be chrysin. The propolis is the exudate of the buds and green parts of *Populus nigra*, and contains the above compound and melezitose. A. Rayner.

Complete saponification of fats in the manufacture of soap base. J. Davidsohn (Chem. Umschau, 1927, **34**, 160—162).—The development of brown spots and rancidity and the loss of perfume in toilet soaps are said to result if more than 0·1% of unsaponified fat is present. In the ordinary boiling operation, since the mass in the pan is not homogeneous, even with long boiling, it is common to find 0·3—0·8% and even more of unsaponified fats present. If the soap is in the right condition this saponification can be completed when the material is only warm, and for this reason slow cooling in frames is preferred in some cases to the quicker machine-cooling method. It is argued that if saponification can be completed under these conditions, it should be possible to get complete saponification much more rapidly by adopting the semi-cold process of soap-making. Various experiments are given confirming this conclusion, and it is shown that tallow with 15% of coconut oil can be completely saponified at 55—60° on a large scale like cold-made soap, by keeping it over-night after saponification has started.

A. Rayner.

Blank test in glycerin analysis by the acetin method (I.S.M.). O. Berth (Chem. Umschau, 1927, **34**, 129—131).—It is suggested that differences in the results of different operators with the acetin method are due mainly to differences in the blank test, and that 50 c.c. of *N*-alkali should be used instead of 5 c.c. In a particular analysis the percentage of glycerol found using 10 c.c. for the blank was 78%, compared with 77·6% using 50 c.c. A true blank can only be obtained using 50 c.c. as in the actual determination, due probably to the influence of carbon dioxide on the titration, as is shown in an experiment in which 10 c.c. of *N*-sodium hydroxide were added to 25 c.c. of water and 125 c.c. of *N*-hydrochloric acid which had been exactly neutralised with *N*-alkali. After boiling the mixture for 15 min. and titrating back, an absorption of 0·46 and 0·38 c.c. was recorded. It is stated further that whilst the I.S.M. method requires acetic anhydride of purity such as to require only 0·1 to 0·2 c.c. of *N*-alkali, such a material is unobtainable. The purest samples were found to require 0·36 to 0·74 c.c. A. Rayner.

Berginisation of fish-liver oils. Ormandy and others.—See II.

Testing viscosity of oils. Schlüter.—See II.

Drying of linseed oil. Wilborn.—See XIII.

Oxyns. Eibner and Munzert.—See XIII.

Patent.

Apparatus for hydrogenation (E.P. 273,045).—See I.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Basic zinc sulphate liquors in lithopone manufacture. W. N. Hirschel (Chem.-Ztg., 1927, **51**, 599).—In the lithopone industry, zinc sulphate liquor is purified by oxidation and subsequent reduction. In these reactions, typical equations for which are given, excess of zinc hydroxide is formed, giving rise to a basic salt that separates from the zinc sulphate liquor after filtration and cooling. This is shown to be $3ZnO,ZnSO_4,8H_2O$. S. S. Woolf.

"Oxyns." V. Oil films from the colloidal point of view. A. Eibner and H. Munzert (Chem. Umschau, 1927, **34**, 183—189, 206—211; cf. B., 1927, 417).—Mainly a theoretical discussion on the relations between the purely chemical and the colloidal reactions involved in the drying of oil and stand oil films. The reaching of the "free from tack" stage is a pseudo-drying due to autoxidation, preliminary to the drying proper which is dependent on the "gelatinising tendency" of the oil, a function of the nature of the individual oil and of the treatment to which it has been subjected. The solubility in alcohol and ether of various fresh and aged oil films was studied, and the drying and the general qualities of an oil are shown to be improved by conversion into stand oil. The insoluble matter in films 6 months old was as follows:—Linseed oil 36%, linseed stand oil 64·5%, tung oil 60%, tung stand oil 86%. The corresponding figures for films at the "pseudo-dry" stage are 28, 50, 15, and 17%. The gel formation essential to true drying proceeds more rapidly in tung oil films than in others owing to the absence from tung oil of mixed glycerides and to its intense tendency to gelatinise. Its claims to be considered as the standard drying oil, in place of linseed oil as hitherto, are advanced. This gel formation is distinct from the formation of semi-solid "polymerised" masses by heating oils in the absence of air, such irreversible gels being absent in ideal dried oil films. From the constants of the portions of aged oil films soluble in water, alcohol, and ether, respectively, the nature of these soluble substances is discussed for the various oils. In the drying of oil films in enclosed vessels, the reversible nature of the gels is demonstrated, the syneresis occurring constituting a suggested "accelerated test." The differences between the drying of tung, linseed, and poppyseed oils are amplified from the above considerations, whilst suitable "colloidal" nomenclature is introduced. S. S. Woolf.

Influence of copper compounds on the drying of linseed oil. F. Wilborn (Farben-Ztg., 1927, **32**, 2542).—Boiled linseed oil containing copper as drier dries more quickly in winter and more slowly in summer than does linseed oil. From laboratory drying experiments it is deduced that variation in humidity rather

than temperature is responsible for the anomalous behaviour. S. S. WOOLF.

Analysis of driers. F. WILBORN (Farben-Ztg., 1927, **32**, 2542).—To avoid the loss of metallic chlorides by volatilisation, when, in the analysis of driers, organic matter is removed by incineration, with subsequent treatment of the ash with hydrochloric acid, also by reduction of the oxides to metal, treatment with strong acid—nitric acid for linoleates, sulphuric acid for resinates—is recommended, especially for lead driers. Experimental details are given. S. S. WOOLF.

Composition of Finnish "fluid resin." E. PYHÄLÄ (Chem. Umschau, 1927, **34**, 145—147, 189—195).—If the soft soap layer separating from the black liquor obtained in cellulose manufacture be decomposed with sodium bisulphate or dilute sulphuric acid, a dark brown, viscous oil ("fluid resin") results. On steam distillation *in vacuo*, this yields 75—80% of "tall oil" and resin crystals, leaving 20—25% of residual pitch. By mechanical separation, 12 fractions varying in viscosity from a mobile oil to a black asphaltic material were obtained from a commercial Finnish "fluid resin." The physical and chemical properties of these are tabulated, and the results of examination of the various fractions are described. The composition of the fluid resin is given as follows:—fatty acids 45·3% (mainly oleic, linolenic, and ricinoleic acids), unsaponifiable oil 2·5%, oily resin acids 21·5%, soft resin acids 10·8%, solid resin acids 13·6%, unsaponifiable pitch 6·3%. S. S. WOOLF.

PATENTS.

Fibrous paint material. G. E. HEYL (E.P. 273,848, 21.4. and 18.5.26).—Paper, wood pulp, spent hops, or similar fibrous material is subjected to a preliminary wet disintegration in an ordinary paper beater, and a subsequent disintegration in a high-speed colloidal grinding machine, binding medium, fillers, and colouring material being incorporated prior to or during either stage of disintegration. Aqueous or oily vehicles may be used at both stages, or the material may be dried after the first stage. S. S. WOOLF.

Binding medium for dyes. E. STERN (G.P. 438,935, 14.10.24).—To water-insoluble fixing media is added 10% or less of a non-swelling carbohydrate, such as formaldehyde–starch, starch compounds of alkalis or alkaline-earths, cellulose, hydrocellulose, or oxycellulose. C. HOLLINS.

Binding medium for dyes. E. STERN (G.P. 439,519, 26.6.25. Addn. to G.P. 438,935, preceding).—As water-insoluble fixing media bitumens, *e.g.*, petroleum pitch, stearine pitch, or viscous mineral oil distillate, are used with the non-swelling carbohydrates of the prior patent. C. HOLLINS.

Method of forming synthetic resinous materials. J. V. MEIGS (E.P. 274,146, 9.2.26).—See U.S.P. 1,593,342; B., 1926, 838.

Mills for grinding paints etc. H. DRYSDALE, and S. SMITH & BLYTH, LTD. (E.P. 274,162, 19.3.26).

Azo dyes and lakes (E.P. 250,909).—See IV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber–filler systems. Colloid problems of the rubber industry. P. STAMBERGER (Kolloid-Z., 1927, **42**, 295—300).—It is found that fillers for rubber, of which the size of the particles lies between 0·5 and 1μ, diffuse through the solvation medium partially or completely with the rubber. The rate of diffusion, and the quantity diffusing with the rubber, depend upon the filler. The view that this behaviour is due to the strongly dispersing action of the rubber is considered to be inadmissible, neither will a purely mechanical explanation meet the facts. It is assumed that the filler is adsorbed on the rubber, the rubber–filler complexes moving together. The effect of carbon black on rubber may also be explained by the adsorption theory. L. L. BIRCUMSHAW.

PATENTS.

Manufacture of an organic selenium compound. Vulcanisation processes. P. I. MURRILL, Assr. to R. T. VANDERBILT CO., INC. (U.S.P. 1,622,534—6, 29.3.27. Appl., [A, B], 30.9.25; [C], 28.8.26).—(A) Compounds of the types $(NRR'\cdot CS\cdot S)_4Se$, $(RO\cdot CS\cdot S)_4Se$, or $(\vdots C\cdot CS\cdot S)_4Se$, *e.g.*, selenium compounds derived from dithiocarbamic, xanthic, dithioacetic, or dithiobenzoic acid, are of value as (B) vulcanisers and (C) accelerators. *Selenium diethyldithiocarbamate*, m.p. 94—95°, is obtained by the interaction of anhydrous sodium selenite (1 mol.), sodium diethyldithiocarbamate (4 mols.), and hydrochloric acid (6 mols.). *Selenium dimethyldithiocarbamate* has m.p. 163—165°. T. S. WHEELER.

Manufacture of a vulcanisation accelerator. H. W. ELLEY and D. H. POWERS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,624,723, 12.4.27. Appl., 2.12.24).—*p*-Nitrosoaryldialkylamines (1 mol.) are condensed with a primary amine (2 mols.), and the product is condensed with a reactive aldehyde (1 mol.) to give compounds of the general formula $R_1R_2N\cdot C_6H_4\cdot N{<}^{NR_2}_{NR_3}{>}CHR_4$, which are of value as accelerators. T. S. WHEELER.

XV.—LEATHER; GLUE.

Analysis of chrome[-tanned] leather and mechanism of the chrome-tanning process. F. HUDSON (J. Soc. Leather Trades' Chem., 1927, **11**, 133—144).—The basicity of the chromium salt on the fibre is obtained from the acid radicals and chromium content. The former are determined by a modification of Meunier and Chambard's method (cf. B., 1923, 367 A). Good agreement has been obtained between this and the nitric acid method (Procter, "Leather Chemists' Pocket Book," 1912, 197), the new method being more rapid. Experiments show that there is no connexion between the basicity of the chromium salt on the fibres of the leather and its ability to withstand the boiling test. The latter is not a satisfactory measure of the completeness of tanning. The results of the test depend not only on the chromium content, but also on the nature of the combined acid radicals. Leather tanned with a basic chromium chloride liquor fails to withstand the test irrespective of its chromium content. Leather tanned with a basic

sulphate liquor to which neutral chlorides have been added shows a lessened stability to the test, the extent of which depends on the concentration of the neutral salt. In leather which withstands the boiling test at the completion of the tanning and fails to do so when finished, the combined bivalent acid radicals originally present have been replaced by univalent acid radicals, *e.g.*, SO_4 by fatty acids. Irregularities in the boiling test are the same for both one- and two-bath chrome-tanned leathers. The different behaviour of combined univalent and bivalent acid radicals is attributed to their different mode of combination. The chromium atom may be attached by primary valency to the carboxyl groups in the collagen or indirectly attached to the imino-groups by means of the bivalent radical. The combination with basic chromium chlorides is of the first type only. The amount of sodium hydroxide accounted for in some determinations by the new method of basicity determination was in excess of that used to the extent of 0·9% on the leather. It is suggested that neutral salts are attached to the chromium atom by secondary valency forces in accordance with Werner's theory.

D. Woodroffe.

[Determination of] insoluble [matter] in tannin solutions. J. G. Parker and J. T. Terrell (J. Soc. Leather Trades' Chem., 1927, **11**, 196—199).—Tests on quebracho, solid oakwood, and chestnut extracts, using the official method of determining the insoluble matter and the Kubelka method (cf. B., 1925, 412, 516, 603), show that the latter indicates a decreased amount of insoluble matter, in some cases to a marked degree; the treatment darkens the colour of the solution both in red and yellow units. The Kubelka method was shown to yield results as concordant as those by the official method. It does not determine the true insoluble matter. D. Woodroffe.

Adhesives and adhesion. McBain and Lee.—See I.

XVI.—AGRICULTURE.

Accumulation and movement of nitrates in a four-field rotation. N. Mokin (Isvestia opitnomu Severnogo Kavkaza [Russia], 1926, No. **9**, 22—35).—On black or clean fallow the quantity of nitrates was large, the greatest accumulation occurring in the autumn. During the summer the nitrate content varied according to rain and tillage; towards the spring the fallow lost most of the nitrate. The course of nitrate accumulation was examined for maize, potato, and winter wheat (following fallow); with winter wheat the nitrates do not disappear at all during the growing season. With maize and potato the nitrate content rises from spring to mid-summer, then decreases until the early autumn, rising again in late autumn. The author concludes that clean fallows do not stimulate nitrate accumulation.

Chemical Abstracts.

Determination of sulphur in arable soil. G. Bertrand and L. Silberstein (Bull. Soc. chim., 1927, [iv], **41**, 950—954).—To determine total sulphur the sample is evaporated to a paste with fuming nitric acid and neutralised with sodium carbonate solution, excess being added and the whole evaporated to dryness and fused with an equimolecular mixture of sodium carbonate and nitrate. The sulphate is dissolved in dilute hydrochloric acid and precipitated by means of barium chloride. Sulphates present as such in the soil are separated by extracting at least three times with boiling dilute hydrochloric acid. Electrical heating is used throughout to prevent contamination from sulphur compounds in burnt gas. F. S. Hawkins.

Modifications of nitrogenous substances in soil dried in the air and left fallow. A. Lebediantzef (Compt. rend., 1927, **185**, 293—295; cf. B., 1924, 305, 436; 1926, 182).—Duplicate samples are taken from the top 20 cm. and 40 cm. layers of soil of triennial crop rotation, and the top 20 cm. layer of fallow soil, and one series is dried as in previous work, the other left undried. All the samples are exposed to the air, left fallow, and kept moist by distilled water, for 180 days. Figures are given for the proportions of nitrate and ammoniacal nitrogen (determined by the methods of Schlœsing and of Boussingault) in the samples at regular intervals. The dried earth is distinguished by a gradual accumulation of nitrates, and by an initial increased content of ammoniacal nitrogen, which subsequently fluctuates.

E. W. Wignall.

Relation between the available phosphoric acid of soils and the geological formation. H. Niklas, R. Pürckhauer, and H. Poschenrieder (Z. Pflanz. Düng., 1927, **A9**, 136—160).—The *Azotobacter* test for phosphoric acid has been applied to some 1300 soils, derived from different geological formations in Bavaria, and certain relationships are found between the content of the soils in available phosphoric acid and their geological origin. In general, soils from marine deposits are less in need of phosphoric acid than those of terrestrial origin. Further, soils with an acid reaction are generally definitely poorer in phosphoric acid than those with a neutral or alkaline reaction, probably because the former have been subjected to more thorough leaching processes. The *Azotobacter* test is very satisfactory for this type of investigation. C. T. Gimingham.

Do soil zeolites contain directly exchangeable hydrogen ions? M. Trénel (Z. Pflanz. Düng., 1927, **A9**, 121—135).—Sodium permutite, deprived of base by continuous extraction with water saturated with carbon dioxide, does not show an acid reaction either in water or in potassium chloride solution. The author holds that, on removal of the bases from soil zeolites, "free combining power" remains, and that, contrary to the views of Hissink and others, there is no direct replacement by hydrogen ions. Only the aluminium adsorbed on the surface of the mineral soil colloids is exchangeable. The injurious action of unsaturated soils on plants is explained by the suggestion that certain plants are not able to obtain sufficient nutrient bases on account of the adsorptive capacity of the unsaturated soil particles. The degree of unsaturation of such soils should be investigated in suspensions in potassium chloride solutions. The "total acidity" and the p_H of a suspension in potassium chloride solution are functionally dependent on one another; hence, it is the exchanged aluminium which brings about the acidity of mineral soils.

C. T. Gimingham.

Determination of soil fertility by chemical

means. K. Bamberg (Z. Pflanz. Düng., 1927, **A9**, 161—175).—Detailed investigations on the determination of phosphoric acid by extraction with different acids are reported, the results being compared with those given by Mitscherlich's physiological method. Special attention was given to the effect of the p_H of the acid extract on the amount of phosphoric acid dissolved, and, in order to obtain the same final p_H in the extracts, the amounts of acid used were adjusted to the buffering capacity of the soils. The phosphoric acid dissolved was found to depend not only on the p_H, but also on the nature of the soil phosphates and on the properties of the particular acid used. Different acids dissolve different amounts of phosphoric acid from the same soil even when the final p_H and other conditions are the same. Much further work on the subject is required.

C. T. Gimingham.

Influence of reaction of soil on formation and composition of marjoram. H. Deel and (Mrs.) Deel (Bull. Soc. chim., 1927, [iv], **41**, 955—957).—The maximum yield was obtained in soil at p_H about 9·5. Variations in the value of p_H caused large variations in the weight of plant and oil obtained per hectare, but only small alterations to their ratio until the p_H value fell below about 5·6. F. S. Hawkins.

Automatic recording device for carbon dioxide in air from 0 to 3·5%. C. Z. Rosecrans (J. Opt. Soc. Amer., 1927, **14**, 479—490).—This instrument has been designed for experimental work in greenhouses, and depends on the thermal conductivity of the gas mixture. An accuracy of 0·05% of the scale reading, independence of scale reading on velocity of gas flow, and compensating devices for current and temperature fluctuation are the principal claims. The application of the instrument to the analysis of other gas mixtures is discussed.

R. W. Lunt.

Relation of size of oil drops to toxicity of petroleum oil emulsions to aphids. E. L. Griffin, C. H. Richardson, and R. C. Burdette (J. Agric. Res., 1927, **34**, 727—738).—The toxicity of oil emulsions to aphids increased with the size of the oil droplets and depended to a far greater extent on this than on the nature of the oil or emulsifier used. With large-drop emulsions a far greater proportion of oil was retained by the leaf surfaces, and presumably by the bodies of the insects. These phenomena are explained on the basis of the intensity of repulsion between the negatively charged oil droplet sand the negatively charged leaf surface.

A. G. Pollard.

Sulphuric acid as a weed spray. A. Åslander (J. Agric. Res., 1927, **34**, 1065—1091).—The influence of various conditions on the killing of weeds by spraying with sulphuric acid (1—2%) has been investigated. Sprayed plants of mustard, kept under different conditions of humidity, were all killed, but most readily in dry air (30% relative humidity). Plants grown in moist soil were killed by a 1·5% solution, whereas in dry soil a 2% solution was required. The plants were killed much more quickly at 30° than at 6°. Spraying with water 1 hr. after treatment with 2% sulphuric acid did not decrease the effect of the acid. Solutions of iron sulphate (5—15%) were much slower in action, and were most destructive under conditions of high humidity and low temperature. Oat plants grown with the mustard were not injured either by the sulphuric acid or iron sulphate solutions. C. T. Gimingham.

Decomposition of vegetable matter under soils. Taylor.—See II.

XVII.—SUGARS; STARCHES; GUMS.

Colour in the sugar industry. I. Colour nomenclature. II. Colorimetric clarification of turbid sugar solutions. H. H. Peters and E. F. Phelps (U.S. Bur. Standards, Techn. Paper 338, 1927, **21**, 261—308).—This paper is the first of a series to be published on the application of precise spectrophotometric methods to the colorimetric investigation of sugar products. In Part I the terms and symbols employed, which are those recommended by the Optical Society of America, are defined and explained. Part II deals with the preparation of solutions of sugar products for colorimetric analysis. The method of preparation usually employed, which consists in making up an aqueous solution of appropriate colour concentration, treating it with kieselguhr, and filtering through a folded filter paper, is unsuitable for precise colorimetric work. Solutions thus obtained are seen to be optically turbid if observed obliquely under strong illumination in a dark room, and their optical properties are liable to change for a considerable time after preparation. Moreover, kieselguhr has selective adsorbent action on the colouring matters present. To avoid these defects the authors have worked out a method of preparing solutions, of which the following is a bare outline. A known weight of the sample is made up to a solution of about 55° Brix by addition of boiling water, added in small quantities. Any further reduction of colour necessary is effected by dilution with a specially prepared solution of pure decolorised sucrose of the same density. After removal of any coarse impurities by straining through cloth, the solution is shaken for 5—15 min. with 2% of dry, specially treated asbestos fibre and filtered. It is finally filtered repeatedly through a pad of similar asbestos in a Gooch crucible, until optically clear. Spectrophotometric observations are made on the prepared solution in a glass cell, a similar cell containing pure water or sucrose solution being used for comparison. If the thickness of the liquid layer in the cell is b cm. and the concentration of the sample is c g. of dry substance per c.c., and T is the transmittancy, *i.e.*, the fraction of the incident light which is transmitted by the solution (divided if necessary by the corresponding fraction for the solvent, which is usually 1), then by the Lambert–Beer law the expression $(-\log T)/bc$ is a constant for the sample and equal to $-\log t$, the specific absorptive index. This represents the absorptive power of unit weight of dry substance of the sample for the particular light employed. It varies with the wave-length, but the authors have found that for a wave-length of 560 mμ it has substantially the same value as for white light. If $-\log t$ for light of this wave-length be divided by 0·00485, the corresponding value for water, the quotient n represents the number of units of colouring matter in 1 g. of dry substance of the sample. J. H. Lane.

Determination of sugar and non-sugars in sugar beet. A. B. FREMEL (Bull. Sakharotresta, 1927, No. 1, 50—66).—The determination should preferably be made on an aqueous extract prepared at 20°.

CHEMICAL ABSTRACTS.

Use of hyposulphites in the [beet] sugar industry. R. MESTRE (Bull. Assoc. Chim. Sucr., 1927, 44, 320—334).—The use of readily hydrolysable hyposulphites, such as those of zinc and aluminium, is recommended for raw juices. The bases liberated by hydrolysis act as adsorbents and precipitants of colloidal impurities, whilst the liberated acid combines loosely with stronger bases, such as lime, or with organic matters, from which it is set free by subsequent sulphitation. Diffusion juice is treated with 1 litre of 10% zinc hyposulphite solution per 20 hectolitres. Data are given showing how the p_H value and viscosity of juices so treated change during the later stages of sugar manufacture. J. H. LANE.

Determination of sugar in carbonatation scums. P. ORTH (Bull. Assoc. Chim. Sucr., 1927, 44, 334—336).—According to Vondrak (B., 1923, 369) and Kunz (B., 1925, 255) the sugar in carbonatation scum cakes can be accurately determined only after complete dissolution of the calcium carbonate. Using acetic acid for the latter purpose, and basic lead acetate for subsequent clarification, the author found differences up to 1·38% in the sugar content of scums, as compared with the usual method. The extent of the differences may depend on the quality of the lime used for defecation. Prolonged contact of scums with water appears to render the whole of the sugar determinable by the usual method. J. H. LANE.

Determination of sulphurous acid in sugar solutions. R. MESTRE (Bull. Assoc. Chim. Sucr., 1927, 44, 317—319).—The determination of sulphur dioxide in sulphited beet factory syrups cannot be satisfactorily carried out by the distillation method owing to the small quantities to be determined and the large excess of sugar. Direct iodometric titration will give consistent results provided the liquids are not alkaline.

J. H. LANE.

Effect of heat on sugar-refinery products. M. I. NAKHMANOVICH and I. F. ZELIKMAN (Nauch. Zapiski [Russia], 1926, 4, 107—115).—Products of high purity, but not those of low purity, are unchanged at 80° for 12 hrs., although at 100° considerable decomposition takes place. CHEMICAL ABSTRACTS.

Purification and drying of potato starch. MARILLER (Bull. Assoc. Chim. Sucr., 1927, 44, 299—303).—The relative merits of vats, tables, and centrifugal apparatus for the purification and deposition of starch, and of hot-air and vacuum apparatus for drying, are briefly discussed, and some types of starch centrifuges and dryers are described with data of output and efficiency. Continuously operating centrifugal machines simplify the processes of purification and deposition, and reduce the labour required. Vacuum dryers are strongly recommended. J. H. LANE.

Albumin from carbohydrates. CLAASSEN.—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Antiseptic action of the bitter substances of hops. W. WINDISCH, P. KOLBACH, and W. SCHÜREN (Woch. Brau., 1927, 44, 285—290, 297—301, 309—314, 324—328, 335—339, 345—350).—The minimum amounts of the different hop resin fractions required to inhibit entirely the development of *Bacillus Delbruckii* in wort under various conditions of hydrogen-ion concentration were determined directly, and also after 2 hrs.' boiling, and after 2 hrs.' boiling followed by fermentation. *B. Delbruckii* was employed because its development could easily be measured by titration of the lactic acid produced. The antiseptic power of the α-bitter acid, humulone, is much greater than that of the other hop resins. It is very dependent on hydrogen-ion concentration, being 30 times as great at p_H 4·3 as at p_H 8·2. Probably the free acid alone is antiseptic, and the salts are indifferent. Boiling for 2 hrs. in wort reduces the antiseptic power, but rather increases its sensitiveness to hydrogen-ion concentration. In wort having the average reaction of beer (p_H 4·3), the amounts of humulone required to inhibit growth in 100 c.c. of wort were :—Before boiling, 0·75 mg. ; after boiling, 0·83 mg. ; and after boiling and fermentation, 1·65 mg. The resin fraction comprising the β-acid, lupulone, plus the soft resins, is much less active and less sensitive to hydrogen-ion concentration. The amounts of humulone required to inhibit growth in 100 c.c. of wort after 2 hrs.' boiling, at a reaction of p_H 7, at the average reaction of wort (p_H 5·6), and at the average reaction of beer, are 14, 5·0, and 0·83 mg., respectively, the corresponding figures for lupulone, plus soft resins, being, 30, 18, and 16 mg. The antiseptic action of the hard resins is negligibly small. J. H. LANE.

Fermentation of pectin substances in mixed cultures. V. L. OMELIANSKII and M. M. KONONOVA (Arkh. Biol. Nauk, 1926, 26, 53—58).—Normal fermentation of retting flax, with easy separation of fibres, takes place on addition of aerobic organisms (*Bacterium fluorescens liquef.*, *B. coli*, *B. mesentericus vulg.*, and *Oidium lactis*) to the anaerobic *Granulobacter pectinovorum*. Hence pure cultures of *G. pectinovorum* should be obtainable from spores in pasteurised flax straw, using aerobic non-sporogenic organisms which are eliminated by subsequent pasteurisation. At the end of flax fermentation the p_H changes from 6·4—6·2 to 4·6—4·2.

CHEMICAL ABSTRACTS.

Nutritive value of spent hops. DAVIES and SULLIVAN.—See XIX.

Albumin from carbohydrates. CLAASSEN.—See XIX.

PATENTS.

Production of sparkling wines. M. JORDT (E.P. 274,707, 11.12.26).

Manufacture of vinegar. A. K. CROAD. From F. NOLDIN (E.P. 274,328, 1.11.26).—See U.S.P. 1,610,465 ; B., 1927, 122.

XIX.—FOODS.

Oiliness in milk. A. T. R. MATTICK (J. Agric. Sci., 1927, 17, 388—391).—A widespread "oily" taint of milk has been traced to the catalytic oxidising action of

exceedingly minute amounts of copper taken up by the milk from the surface of the cooler or other apparatus. The reaction is dependent on free access to oxygen, and occurs only at comparatively low temperatures. The action of bacteria limits the production of "oiliness" to cold weather, either by absorption of oxygen, or by the production of acidity. "Oiliness" is not identical with other flavours in milk caused by somewhat larger amounts of copper. C. T. GIMINGHAM.

Factors, other than bacteria, which influence the body of buttermilk. G. KNAYSI (J. Agric. Res., 1927, **34**, 771—784).—In the preparation of artificial buttermilk the addition of sodium salts improved the homogeneity and "body" of the product. This effect depended to some extent on the particular salt used. Heating to 100° resulted in a better product than the customary heating to 80°, the improvement depending on physical conditions and not on the removal of micro-organisms. The tendency of artificial buttermilk to separate when stored at high temperatures is largely a physical phenomenon, and was not affected by the addition of such substances as starch and gelatin.
A. G. POLLARD.

Movement of substances through cheese. Theory of salting-down cheese. M. YEGUNOV (Visti Odeskii Silsko-Gospodar'skii Inst., 1925—6, **1**, 90—93).—Various kinds of cheese were tested with copper sulphate and silver nitrate as diffusion solutions, and the relation between the height of diffused substance and the square root of the time was determined.
CHEMICAL ABSTRACTS.

Production of food albumin from carbohydrates. H. CLAASSEN (Chem.-Ztg., 1927, **51**, 577—578).—A dried yeast containing 42% of albumin can be obtained after development of the organism by means of molasses, ammonium sulphate, and superphosphate, but it cannot compete with natural feeding stuffs owing to its high cost of production, which is approximately twice that of dried yeast from brewery residues and five times that of nut cakes with the same albumin content. The use of cheaper sources of carbohydrates such as waste waters from cellulose, starch, or sugar factories, or the substitution of other micro-organisms for yeast, give unsatisfactory results. F. R. ENNOS.

Comparison of meats pickled with saltpetre made from Chili saltpetre and from synthetic sodium nitrate. G. RIESS and R. MEYER (Chem.-Ztg., 1927, **51**, 361—363).—No difference could be detected in the colour, odour, or taste of meat pickled with salt and saltpetre, when the latter was made from natural or from synthetic sodium nitrate. Determination of sodium chloride in the outer and inner layers after definite times indicated that the rate of penetration of salt into the meat was not appreciably different in the presence of the two nitrates. The process of pickling and formation of the red colour are accompanied by a gradual increase in the proportion of nitrite present.
F. R. ENNOS.

Lowering of the "essence titer" in preparations of black mustard seeds. LASAUSSE (Ann. Falsif., 1927, **20**, 275—279).—Determinations were made of the allyl mustard oil in mustard flour and other mustard preparations, after keeping for varying periods in closed vessels, by the following reliable method: 5—20 g. were macerated with water and alcohol, and 75—85% of the liquid was distilled very slowly (3 hrs.) from an oil-bath at 130° into standard ammoniacal silver nitrate solution. The distillate was finally heated in a water-bath under a reflux condenser, and the precipitated silver sulphide collected and washed. The excess of silver in the filtrate was then determined with $0 \cdot 1N$-thiocyanate solutions (cf. Colombier, B., 1926, 606). A gradual diminution in the essence content was observed, depending on the time the preparations were kept and the nature of the containing vessel. F. R. ENNOS.

Composition of fenugreek seed and the inconvenience of its mixture with grain destined for milling. E. FLEURENT (Compt. rend., 1927, **184**, 1344—1345).—From a detailed examination the author's previous conclusions are verified (cf. B., 1926, 418). Apart from its bitterness, the extracted resin gives out an acrid, penetrating odour which is also possessed by the aqueous extract of the grain. E. H. SHARPLES.

Net energy values of maize silage, soya-bean hay, lucerne hay, and oats. E. B. FORBES, W. W. BRAMAN, M. KRISS, J. A. FRIES, C. D. JEFFRIES, R. W. SWIFT, R. B. FRENCH, and J. V. MANCHER, JUN. (J. Agric. Res., 1927, **34**, 785—796).—The net energy values of these feeding stuffs were determined by direct calorimetry. The heat values were more nearly proportional to the dry matter present than to the metabolisable energy of the foods; the latter in turn depended on the nature of the material, notably whether bulky or concentrate. A. G. POLLARD.

Nutritive value of dried spent hops. W. L. DAVIES and R. S. SULLIVAN (J. Agric. Sci., 1927, **17**, 380—387).—Dried spent hops contain crude protein 21·5%, ether extract 8·3%, crude fibre 25%, nitrogen-free extractives 33%, ash 6·2%, and water 5·8%. The digestibility is low, and the material is not readily eaten by sheep. The production starch equivalent was 24·5. Attention is drawn to the use of spent hops to absorb molasses or treacle. C. T. GIMINGHAM.

Influence of binding material in sausages on the detection of added water by the Feder method. H. WILLEKE and F. JUNKER (Z. Unters. Lebensm., 1926, **52**, 478).—In determining the Feder ratio for sausages, differences were observed in the numbers for fat-free organic matter and nitrogenous matter. This was traced to the use of protein substances as binding material. The presence of 3—5% of such binding material effectively conceals considerable additions of water as determined by the Feder process. A. G. POLLARD.

Vanilla powders. J. MOROY (Ann. Falsif., 1927, **20**, 21—25).—Vanilla sold as powder is necessarily prepared by grinding with sugar; the quantity of sugar present and, consequently, the proportion of vanilla (to within 1—2%) may be determined by means of the saccharimeter, provided that a microscopical examination reveals the absence of any insoluble matter foreign to vanilla. D. G. HEWER.

Occurrence of arsenic and lead on fruit after spraying. K. LENDRICH and F. MAYER (Z. Unters.

Lebensm., 1926, **52**, 441—457).—Fruit from sprayed trees retained varying amounts of lead and arsenic on the surface. No indication of penetration into the pulp of the fruit was observed. The presence of lead in addition to arsenic is objectionable as its toxicity is cumulative. With fruit, other than apples and pears, where washing to remove spray material is not practicable the use of calcium in place of lead arsenate in sprays is suggested. In most cases spraying should be carried out once only, and this soon after the flowering period. Analytical methods for determining arsenic and lead are examined. A. G. POLLARD.

Discoloration of canned cranberries. F. W. MORSE (J. Agric. Res., 1927, **34**, 889—892).—The brown or black discoloration appearing in tins of cranberry sauce was traced to the reaction of iron dissolved from the inside of the can with the colouring matter of the fruit and to a small extent with the tannin. A. G. POLLARD.

Straw. HONCAMP.—See V.

PATENTS.

Preservation of natural products [eggs etc. with wax]. S. FOWLER and E. EDSER (E.P. 274,200, 16.4.26).

Evaporating apparatus (E.P. 272,703).—See I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of higher alcohols, particularly hydrogenated phenols, in presence of some organic solvents, especially hydronaphthalenes. II. Determination of methylhexalin in presence of tetralin. K. LINDNER and J. ZICKERMANN (Chem. Umschau, 1927, **34**, 199—205).—The methods described earlier (cf. B., 1924, 768) are invalidated owing to the fact that commercial hydrophenols are not chemical individuals, commercial methylhexalin, *e.g.*, being a mixture of the three isomeric hexahydrocresols with ketones and unknown condensation products. A method for determining methylhexalin in the presence of tetralin depends on the distribution of the former between the latter and water. If y be the amount of methylhexalin found in the water layer when 15 g. of a mixture of methylhexalin and tetralin are shaken with 300 g. of water, then x, the percentage content of methylhexalin in the mixture is given by the equation $y^{1\cdot3}=0\cdot76x$. In practice the mixture is obtained free from impurities by steam distillation, the methylhexalin in the aqueous layer being extracted with ether and recovered, whilst that in the tetralin layer is shaken out with water and determined as above. S. S. WOOLF.

Digitonin—its properties, isolation, and determination. I. S. MELLANOFF (Amer. J. Pharm., 1927, **99**, 390—401).—Gravimetric methods (precipitation with cholesterol, α- and β-naphthol, thiophenol, terpineol, and bromo-β-naphthol) for the determination of digitonin are described. Whilst the α- and β-naphthol methods are the most economical, the most accurate results are obtained by precipitation with cholesterol, which forms the least soluble additive compound. The additive compounds of all the above substances with digitonin are separated into their components by means of hot xylene or benzene. The properties and extraction of digitonin from *Digitalis purpurea* are described, and the determination of digitonin showed that there is about 1·4% in the seeds, 31·1% in digitalin (Merck), and 81·1% in digitonin (Merck). E. H. SHARPLES.

Assay of podophyllum. L. E. WARREN (J. Assoc. Off. Agric. Chem., 1927, **10**, 272—280).—The resin content of podophyllum has been determined by three methods, U.S.P. X, U.S.P. IX, and that proposed by Jenkins (cf. B., 1914, 885). The first method consists in digesting and percolating the drug for 3 hrs. with boiling alcohol, an aliquot portion of the tincture being then shaken with chloroform and a saturated solution of potassium citrate. The chloroform layer is then drawn off, the solvent evaporated, and the residue dried and weighed. In the U.S.P. IX method, the drug is macerated for two days with cold alcohol, the extract being evaporated down to a syrup, and the resin precipitated by pouring into cold water slightly acidified with hydrochloric acid, from which it is filtered, dried, and weighed. The Jenkins method consists essentially in extracting with alcohol, shaking an aliquot portion of the extract with chloroform and very dilute hydrochloric acid, followed by further extractions with chloroform–alcohol mixture. The assay given by the first method always exceeded those given by the other methods, in some cases by as much as 70%. The resin obtained by this method also contains an alcohol-insoluble fraction, which is physiologically inert, the ash content is much higher than the limit specified by the U.S. Pharmacopœia, whilst the podophyllotoxin content as determined by the Eder and Schneiter method (cf. B., 1926, 384) is lower than that of the commercial resin. This process is therefore concluded to be unreliable, whereas the Jenkins method is satisfactory. H. J. DOWDEN.

Ephedrine sulphate. ANON. (J. Amer. Med. Assoc., 1927, **88**, 924—925).—Three commercial specimens gave the following values, respectively: m.p. 236—243°, 233°, 230°; α_D in water $-29\cdot27°$, $-26\cdot12°$, $-5\cdot97°$; sulphate 22·76, 28·08, 21·87%; base 76·18, 75·16, 71·64%; α_D of free base in ethyl alcohol, $-6\cdot06°$, $-4\cdot5°$, $+7\cdot5°$. Suggested standards are: base 74·5—77·25%, sulphate 21·8—23·1%, α_D of base $-6\cdot0°$ to $-7\cdot5°$ in absolute ethyl alcohol. CHEMICAL ABSTRACTS.

Ethereal oil from the stems and roots of *Pinus silvestris* and comparison of it with the wood and needle oils of this species of pine. F. W. SEMMLER and H. VON SCHILLER (Ber., 1927, **60**, [B], 1591—1607).—The oil was obtained by distillation of the stems of the German pine with steam and purified by a second steam distillation. It contained a little α- and β-pinene, mainly d-Δ^3-carene, together with d-Δ^4-carene and α-terpinene in quantity which varied with the temperature employed during the preparation. The oil contained also decomposition products of cellulose, and lignin, fatty acids, and abietic acid. d-Δ^3-Carene has b.p. 165—172°, d^{20} 0·8563, n_D 1·4737, $\alpha_D+14°\,45'$. It is converted by ozonisation and subsequent reduction of the ozonide by zinc dust into the *keto-aldehyde*, $CMe_2\left\langle\begin{array}{l}CH\cdot CH_2\cdot CHO\\ |\\ CH\cdot CH_2\cdot COMe\end{array}\right.$ b.p. 125—130°/12 mm., d^{20} 1·013, n_D 1·4651, $\alpha_D+3°\,40'$ [*disemicarbazone*, m.p. 198—199°

(decomp.)], which is oxidised by permanganate to the *keto-acid*, $C_{10}H_{16}O_3$, b.p. 175—182°/12 mm., d^{20} 1·045, n_D 1·4691 (*semicarbazone*, m.p. 182—183°); the corresponding *methyl* ester, b.p. 132—136°/13 mm., d^{20} 1·019, n_D 1·5021 (*semicarbazone*, m.p. 108—110°), and *ethyl* ester, b.p. 140—142°/12 mm., d^{20} 1·000, n_D 1·4531 (*semicarbazone*, m.p. 150—151°), are described. The ketonic acid is transformed by bromine in alkaline solution into carbon tetrabromide, bromoform, and a *dicarboxylic acid*, $C_9H_{14}O_4$, m.p. 112—113° (*silver* and *copper* salts; *methyl* ester, b.p. 145—150°/13 mm., d^{20} 1·076, n_D 1·4571). Oxidation of the keto-acid with permanganate affords *cis*-caronic acid, m.p. 174—175°. The presence of d-Δ^4-carene in the carene fraction of the oil is established by oxidising the latter with potassium permanganate in anhydrous acetone (cf. Simonsen and Rau, J.C.S., 1922, **121**, 1643) and isolation of 1:1-dimethyl-2-γ-ketobutyl*cyclo*propane-1-carboxylic acid as the semicarbazone. The neutral products obtained simultaneously (derived from d-Δ^3-carene) contain the *ketone* $CMe_2<\begin{matrix}CH\cdot CH_2\cdot CMe \\ | \qquad\qquad \| \\ CH\cdot CO\cdot CH\end{matrix}$ b.p. 119—123°/11 mm., d^{20} 1·020, n_D 1·498, α_D — 5° 25′ (*semicarbazone*, m.p. 159—161°), which is reduced by sodium and alcohol to the corresponding, saturated, secondary *alcohol*, $C_{10}H_{18}O$, b.p. 103—107°/11 mm., d^{20} 0·9481, n_D 1·4770, oxidised by chromic acid in glacial acetic acid to the saturated *ketone*, $C_{10}H_{16}O$, b.p. 88—100°/11 mm., d^{20} 0·9262, n_D 1·4760 (*semicarbazone*, m.p. 223—224°). Silvestrene does not appear to be present in the original oil. It is obtained by inversion of d-Δ^4-carene, whereas d-Δ^3-carene can be inverted only with difficulty. The action of hydrogen chloride leads to the formation of a mixture of the dihydrochlorides of silvestrene and dipentene from which the corresponding hydrocarbons are obtained by removal of hydrogen chloride. Other pine oils show close analogy with the root oil, although the pinene content preponderates slightly. The presence of d-Δ^3-carene in the needle oil of *Pinus silvestris* could not be established, but confirmation of this conclusion is desirable. The German pine root oil of turpentine is considered as valuable for technical application as the imported oil except for the preparation of artificial camphor. H. WREN.

Roumanian caraway oil. E. KOPP (Pharm. Zentr., 1927, 68, 212—216).—Crops were grown from caraway seed of Dutch and cultivated Roumanian origin. The seeds obtained, together with those from the wild Roumanian plant (all ground before steam-distillation), showed the following oil content:—Dutch, 3·93%, d^{15} 0·9150, α_D (in 100 mm. tube) + 75·5°, n^{20} (white light) 1·4865, carvone content 65·3%; Roumanian, 5·23%, d^{15} 0·9085, α_D + 78·7°, n^{20} 1·4846, carvone content 61·6%; wild Roumanian, 3·86%, d^{15} 0·9184, α_D + 75·65°, n^{20} 1·4862, carvone content 65·1%. The carvone content of the seeds was 2·57%, 3·22%, and 2·51%, respectively. The cultivated Roumanian seeds, on distillation without grinding, yielded 3·63% of oil (carvone content 73·7%), the residue on grinding and distillation yielding a further 0·61% of oil (carvone content 20%). B. FULLMAN.

Metaldehyde. LEW.—See II.

PATENTS.

Making peroxides of organic acids. R. H. MCKEE (E.P. 271,725, 26.10.26).—In the preparation of acyl peroxides from acid chlorides and a solution of hydrogen or sodium peroxide, the yield is improved by keeping the p_H of the liquid at about 6—8·5 by adding a buffer substance, *e.g.*, sodium monohydrogen phosphate, the temperature being maintained at 1—18°. With a 5% hydrogen peroxide solution, sodium monohydrogen phosphate is added either in such quantity that during the reaction about 90% of it is converted into the dihydrogen compound, or smaller amounts of this or other buffers may be used, sodium hydroxide or soda ash being added during the reaction. B. FULLMAN.

Production of alkylenediguanidines. M. HEYN (E.P. 274,259, 18.6.26. Addn. to E.P. 272,686; B., 1927, 619).—Alkylenediamines containing more than five carbon atoms react with salts of methyl*iso*thiocarbamide in concentrated aqueous solution at 100° to give alkylenediguanidines. *Hexa-* and *deca-methylenediguanidines* are described. C. HOLLINS.

Manufacture of 1-phenyl-3-methyl-5-pyrazolone. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (E.P. 274,366, 5.2.27).—1-Phenyl-3-methyl-5-pyrazolidone is oxidised in hydrochloric acid solution by air or oxygen in presence of a heavy metal salt (ferric chloride, cupric chloride). C. HOLLINS.

Extraction of total alkaloids from ergot. W. STRAUB (G.P. 439,042, 1.2.25).—Ergot is shaken with 45—60% aqueous acetone for several hrs. at 15°. On removing the acetone in a vacuum from the filtrate, the active alkaloids are precipitated practically fat-free and free from amine bases. C. HOLLINS.

Organic selenium compound (U.S.P. 1,622,534).—See XIV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic emulsion. I. Comparison of emulsions made with different bromides. B. H. CARROLL and D. HUBBARD (J. Physical Chem., 1927, **31**, 906—921).—Emulsions of the rapid type have been prepared with potassium, ammonium, sodium, calcium, cadmium, and zinc bromides, and have been compared sensitometrically and as to grain size. Data on the solubility of silver bromide in the unwashed emulsions, on the p_H, and on the viscosity of gelatin in the presence of the various bromides are discussed in relation to the mechanism of emulsion making. L. S. THEOBALD.

Schwarzschild effect. I. LÜPPO-CRAMER (Phot. Ind., 1927, 337—339).—Pure silver bromide diapositive plates were exposed (*a*) to blue light for a short time, (*b*) to yellow light for a long time, the times being so chosen that the same threshold values were obtained in the two cases. On development, (*a*) gave high density and high gamma, whilst (*b*) gave only a thin image and a much lower gamma. Treatment of the plates before exposure with sodium nitrite or sodium formate resulted in no change in the blue sensitivity, but the yellow sensitivity increased, becoming almost equal to that of

the blue. The differences between (a) and (b) may be due to the Schwarzschild effect, in which event it should be influenced by bromine acceptors. This was found to be the case, for after bathing in sodium nitrite, sodium sulphite, sodium bisulphite, sodium formate, phenol, or pyrocatechol, there was practically no Schwarzschild effect. Solarisation and the Herschel effect show that during exposure there is a regressive tendency. It is assumed that this tendency also exists during normal exposure, but since the combination of bromine with silver takes time, this tendency will be shown more at low intensities than at high. It is further supposed that the first atoms of silver which form on exposure can very readily recombine with bromine, and this combination will have more time to take place at weak intensities than at strong, since with the latter the nucleus grows rapidly and becomes less easy to brominate.
W. Clark.

Schwarzschild effect. II. Lüppo-Cramer (Phot. Ind., 1927, 363—365).—The Schwarzschild effect is decreased or prevented by bathing the plate in water and drying. This is not due to the washing out of potassium bromide, but is probably an effect of swelling and re-drying. It is not noticeable at low intensities of exposure, nor if the treatment is given after exposure, and is not a development effect. It is given only with slightly ripened diapositive emulsions. Ordinary plates do not show it, although they show the ordinary Schwarzschild effect. The Herschel effect is affected by water in much the same way as the Schwarzschild effect, indicating that the nuclei are more readily attacked before than after water treatment. The water effect may be due to some sort of agglutination ripening, by which the nuclei become less susceptible to attack in the more compact medium. W. Clark.

Schwarzschild effect as a regression phenomenon. Lüppo-Cramer (Phot. Ind., 1927, 495—498).—A rapid emulsion was pre-exposed to give the maximum density on development, and then given further scale exposures at differing values of It. At the longer exposures and lower intensities the solarisation was very much greater than at the short exposures at high intensity. The preparation of a silver iodide–gelatin emulsion is described which does not show the "water-effect," but which gives a marked Schwarzschild effect, which is prevented by impregnation with sulphite or potassium ferrocyanide, and almost completely so by nitrite. With physical development there is practically no Schwarzschild effect, as is also the case with silver bromide emulsions. Silver bromide–collodion emulsions show a Schwarzschild effect of the same order as fine-grained gelatin emulsions. If the Schwarzschild effect is due to regression, this result may be taken as indicating that gelatin is not a halogen acceptor. In presence of nitrite the latent image on diapositive plates formed by exposure to long-wave-length light begins to print out sooner than when white light of low intensity is used. Evidence is given that the time effect may influence photochemical reactions so strongly that complete reversal may take place. W. Clark.

Time effect. Lüppo-Cramer (Phot. Korr., 1927, **63**, 103—106).—It is shown that bleaching of pre-exposed plates, bathed in phenosafranine bromide or plain Pinacryptol Yellow, followed by a second exposure, is more marked if the last exposure is long and at low intensity than if it is short and at high intensity. This would be expected if the Schwarzschild effect is due to regression having time to take place at the long exposures and low intensities. The time effect can also be observed in absence of dyes, when reversal is carried out by Renwick's method of pre-exposure, iodising, and then giving an ordinary exposure. It is stated that solarisation is also increased when, for the same value of It-I is weak and t is long. The time effect does not appear to play a part in the Herschel effect, although this does not show that the Herschel effect is not a variant of solarisation due to regression. W. Clark.

High-temperature drying of photographic layers. O. Papesch and M. Lippermayr (Phot. Ind., 1927, 193—195).—A general account of a process for the quick drying of gelatin films at high temperatures and low pressures, with a discussion of the advantages to be gained by the use of the method. W. Clark.

Patents.

Preparation of a [coloured] photographic image. W. V. D. Kelley, Assr. to Prizma, Inc. (U.S.P. 1,628,248, 10.5.27. Appl., 19.11.19. Renewed 21.10.26). —A transparent plate, coated with sensitive emulsion on both sides, is exposed on both sides through a screen of opaque and transparent lines, so that unprinted portions on one side correspond with printed portions on the other. The exposed portions are then developed and treated so that they are capable of being dyed. Two-colour selection negatives, taken by known methods so as to record both ends of the spectrum, are then employed, one to each side, to print the prepared positive, which is finally developed and treated with complementary dyes, one to each side. T. S. Wheeler.

Production of a photocollographic printing plate. M. de'Sperati, Assr. to Argentographica (U.S.A.), Ltd. (U.S.P. 1,625,714, 19.4.27. Appl., 12.5.26. Conv., 3.5.25).—The printing plate comprises a celluloid base coated on one side with layers of gelatin, those next to the support being impervious to moisture. The outer layers are sensitised with ammonium dichromate, the prepared plate being exposed to the action of light and used for lithographic printing. T. S. Wheeler.

Laminated [photographic] film with a layer of polymerised vinyl chloride. R. L. Stinchfield, Assr. to Eastman Kodak Co. (U.S.P. 1,627,935, 10.5.27. Appl., 1.4.26).—A flexible photographic film comprises a layer of polymerised vinyl chloride, coated on both sides with films of cellulose acetate, one of which carries a sensitive emulsion. The cellulose acetate inhibits accumulation of electric charges on the polymerised vinyl chloride.
T. S. Wheeler.

Photographic printing in colours. L. Dufay (U.S.P. 1,631,524, 7.6.27. Appl., 25.1.24).—See E.P. 217,557; B., 1925, 379.

Kinematograph and other colour films. J. E. Thornton (E.P. 274,591, 6.5.26).

XXII.—EXPLOSIVES; MATCHES.

"Force" and other constants of explosives. N. YAMAGA (J. Fac. Eng. Tokyo, 1927, **17**, 79—88).—On the assumption that thermodynamically the gas reaction at the moment of explosion of smokeless powders may be represented by the water-gas reaction (cf. B., 1925, 301) it is shown that the "force," the explosion temperature, and the specific volumes of the explosion gases may be obtained by graphic solution of three equations if the composition of the powder and the heats of formation of the constituents are known. Cordites with 15—65% of nitroglycerin are taken as examples. In the case of a nitrocellulose powder the effects of variations in the diphenylamine and volatile matter contents on the explosive constants are investigated. S. BINNING.

Determination of moisture in smokeless powder. E. BENESCH (Chem.-Ztg., 1927, **51**, 578).—A current of dry air is drawn over weighed portions of the powder in two glass tubes heated for 4 hrs. in a water bath. One tube is connected to an absorption apparatus containing sulphuric acid for determination of the total volatile matter, which consists of moisture, alcohol, and acetone. Following the other is a combustion tube containing red-hot copper oxide, whereby the alcohol and acetone are burnt to water and carbon dioxide, which, with the moisture originally present, are absorbed and weighed in the usual manner. From the weights of total volatile matter, total moisture, and carbon dioxide the percentages of moisture, alcohol, and acetone in the powder can be calculated.
F. R. ENNOS.

Explosive ammonium salts. H. KAST (Z. ges. Schiess- u. Sprengstoffw., 1926, **21**, 205—209; 1927, **22**, 6—9, 30—34, 56—61, 77—80, 99—102, 131—135).—A summary is given of the literature on the formation and properties of the following explosive ammonium salts: azide, nitrite, nitrate, chlorate, perchlorate, permanganate, dichromate, and trichromate. Values are given for the heats of formation and explosion, explosion temperature, specific energy, detonation velocity, brisance, and sensitiveness to shock, friction, and heat. In investigating the explosive properties of ammonium nitrate it was found that complete detonation could not be obtained, nor could a definite detonation velocity be found by either the chronographic or Dautriche methods. With mixtures of ammonium nitrate and sulphate the detonation velocity varied with the cubic density of the charge, being, for the 60/40 mixture, 1900 and 2430 m./sec. for cubic densities of 0·88 and 0·90, and, for the 70/30 mixture, 2600 and 2860 m./sec. for cubic densities of 0·76 and 0·87. Ammonium perchlorate is not so hygroscopic as the nitrate, but more hygroscopic than potassium chlorate. Ammonium perchlorate is more stable in hot storage than ammonium nitrate is, and the decomposition is hastened by the presence of the oxy-chlorine decomposition products. Comparative data are given for the ignition points of potassium chlorate and ammonium perchlorate and nitrate, both alone and in presence of certain hydrocarbons, metals, metallic compounds, and combustible organic compounds. Mixtures of potassium permanganate and ammonium nitrate may interact to form the very sensitive and explosive ammonium permanganate. No definite dividing line can be drawn between explosive and non-explosive mixtures of ammonium nitrate with other ammonium or alkali salts, as the mode of ignition, degree of confinement, and size of grain are important factors. The use of blasting to break up mixtures of ammonium salts that have set hard is deprecated. S. BINNING.

Stability of [per]chlorate explosives on storage. VANDONI (Mém. Poudres, 1927, **22**, 150—158).—The explosive properties of explosive P (ammonium perchlorate 61·5%, sodium nitrate 30%, paraffin 8·5%) are known to deteriorate on storage to such an extent that re-working of the explosive becomes necessary, although the absorption of moisture is small. The loss of strength cannot be explained by simple hydration, but is due to a slow recrystallisation of the milled salts under the influence of the absorption of moisture, the amount of which is determined by the sodium nitrate content. S. BINNING.

Reduction of the viscosity of CP2 by boiling in alkaline waters. KOVACHE (Mém. Poudres, 1927, **22**, 159—173).—The reduction of the viscosity of some war-stocks of CP2 (soluble nitrocellulose) from a value of 700—1300 to the stipulated acceptance value of 200 was found to require the boiling of the powder for 36—45 hrs. The operation could be much shortened by adding certain alkaline compounds to the water. The ascending order of effectiveness was the carbonates of calcium, sodium, potassium, and ammonium, and ammonia. With sodium carbonate the addition of 10 kg. per 1000 kg. of CP2 reduced the boiling operation to 16 hrs., whilst addition of 30 kg. reduced it to 8 hrs. No alteration in the nitrogen content of the powder was noticed, and its stability was not affected. The loss of powder was no greater than occurred during the prolonged boiling with ordinary water. The rate of reduction of viscosity increased with the amount of salt employed except in the case of calcium carbonate, where it remained constant. S. BINNING.

Liquid-air blasting explosives. C. BUNGE (Z. ges. Schiess- u. Sprengstoffw., 1927, **22**, 21—27).—Stephan's views on the relative liabilities of ordinary and liquid-air explosives to cause accidents are controverted. The claims of the Sprengluft A.-G. that their "oxyliquit KS0" and "oxyliquit KS7" can be used with safety in dusty mines are not confirmed by experiments that are described. The latter cartridge gives on explosion fumes containing compounds of oxygen and chlorine which are injurious to health. S. BINNING.

Hydrogen peroxide explosives. M. BAMBERGER and J. NUSSBAUM (Z. ges. Schiess- u. Sprengstoffw., 1927, **22**, 125—128).—Attempts have been made to produce explosive mixtures in which hydrogen peroxide is the oxygen carrier. Paraformaldehyde mixed with hydrogen peroxide (60% strength) formed a paste which detonated when heated, or when brought into contact with lead. From this paste crystals of m.p. 50° were obtained which were sensitive to shock and friction, and gave an expansion of 600—630 c.c. in the lead

block test. With cellulose a higher concentration (83·4%) of peroxide is required, and dissolution is assisted by treating the cellulose with alkali. 200 g. of peroxide mixed with 40 g. of wood cellulose gave on explosion expansions of 408—414 c.c., but in 48 hrs. the value fell to 349—371 c.c. Tests in a quarry with a mixture of hydrogen peroxide (88—90% strength) 76 pts., cotton wool 15, and vaseline 9 gave results equal to those from dynamite No. 1, but the high cost and small supplies of hydrogen peroxide render the use of these explosives unpractical. S. Binning.

Method for testing initiating substances (detonators). L. Wöhler, J. F. Roth, and K. Ewald (Z. ges. Schiess- u. Sprengstoffw., 1927, **22**, 95—99, 135—139).—The effects are discussed of certain modifications in the technique of a method previously described for comparing the strengths of detonators (*ibid.*, 1925 and 1926). S. Binning.

Mixed acids. Loriette and Jovinet.—See VII.

Patent.

Propellant powder [for shot-guns]. J. M. Olin and A. S. O'Neill, Assrs. to Western Cartridge Co. (U.S.P. 1,627,859, 10.5.27. Appl., 4.4.24).—A dense colloided nitrocellulose powder is ground and rumbled with dinitrotoluene in presence of water for 2 hrs. at above 75°. The grains are then rapidly cooled and dried. The treatment with dinitrotoluene is repeated at 80° for ½ hr. The product has approximately the density of black powder, and is mixed with about 10% of it to form a propellant suitable for use in shot-guns. T. S. Wheeler.

Manufacture of a [shot-gun] propellant powder. A. S. O'Neill, Assr. to Western Cartridge Co. (U.S.P. 1,627,861—2, 10.5.27. Appl., [A] 10.2.23; [B] 4.9.23).—(A) Dense colloided nitrocellulose treated as described in U.S.P. 1,627,859 (preceding abstract) is employed as a propellant for shot-guns without addition of black powder. (B) Dense colloided nitrocellulose, *e.g.*, waste ammunition, is flaked in presence of water and then rumbled with dinitrotoluene as in (A), in presence of a solution of potassium nitrate. T. S. Wheeler.

Manufacture of an explosive powder. A. S. O'Neill, Assr. to Western Cartridge Co. (U.S.P. 1,627,863, 10.5.27. Appl., 1.12.24).—Molten ammonium nitrate is dropped through a sieve into paraffin oil or other waterproofing material, and is obtained in the form of a coated spherical granule, of value in the manufacture of explosives. Alternatively, the spherical granules are rumbled with a polymerisable oil, *e.g.*, China wood oil, in a volatile solvent, *e.g.*, carbon tetrachloride. T. S. Wheeler.

XXIII.—SANITATION; WATER PURIFICATION.

Treatment of water to prevent corrosion. J. R. Baylis (Ind. Eng. Chem., 1927, **19**, 777—781).—With most waters having p_H 6·5 or under, zinc galvanising corrodes rapidly, but where it is 7·5 and over the galvanising has a long life. Coal-tar coatings are not durable in moderately corrosive water since they are probably slightly porous, but with non-corrosive water they remain satisfactory after 25 years. Portland cement mortar is one of the best coatings for pipes at ordinary temperatures or up to 60—70°, and the lining is best applied centrifugally. Corroding iron removes all the free carbon dioxide in water and nearly half of the combined carbon dioxide, the calcium carbonate precipitated thereby materially aiding the formation of an impervious coating. The majority of domestic waters being below the saturation point of calcium carbonate, corrosion is best prevented by raising the amount to saturation equilibrium, which depends upon the p_H value, and to a certain extent upon other salts in solution. If the calcium carbonate is less than 25 pts. per million, lime may be added, but if above that figure sodium carbonate or sodium hydroxide is used. Water saturated with calcium carbonate offers a fairly good resistance to corrosion; if supersaturated, lime incrustation results. W. G. Carey.

Recarbonation of softened water. C. P. Hoover (Ind. Eng. Chem., 1927, **19**, 784—786).—Water softened with lime is supersaturated with normal carbonates of calcium and magnesium the deposition of which on the sand filters, in distribution systems, meters, and hot-water installations is prevented by adding carbon dioxide before filtration. The use of flue gases for this purpose imparts taste to the water and fouls the compressor with condensed hydrocarbons, but fuel oil, natural gas, coke, and pulverised coal have also been used in suitable furnaces. For a small plant, gas or petroleum is the most suitable fuel, both giving tasteless and odourless products containing about 12% CO_2, compared with 4% when coke is burnt in an ordinary furnace. Producer gas made from coke is the most economical fuel for large installations. W. G. Carey.

Manganese in waterworks. C. A. H. von Wolzogen Kühr (J. Amer. Water Works Assoc., 1927, **18**, 1—31).—Complete removal of manganese dissolved in the raw dune water supplying the purification works at Leiduin, Amsterdam, takes place in the rapid and sand filters by oxidation into insoluble oxide, and by the same process a part of the manganese settles in the shallow canals of the drainage area. The deposit in the filters etc., as determined by iodometric and oxidimetric titration methods, consists of manganese dioxide. The oxidation is due to bacterial action. Chemical oxidation cannot take place owing to the low alkalinity (p_H 8·1) of the raw dune water. Isolation from raw dune water, filter mud, etc. of a group of bacteria capable of oxidising manganous compounds has been effected. The media used consisted of dune water, 100 c.c.; agar, 2·5 g.; calcium acetate, 1·0 g.; ammonium sulphate, 0·1 g.; dipotassium phosphate, 0·05 g.; manganese carbonate, 1·0 g. The colonies developed possessed centres usually dark brown to black in colour, according to the degree of manganese oxidation. Colonies developed on faintly acid media also were dark in colour. The colouring material was found to be manganese dioxide. The bacteria occurred as mono- and diplo-cocci, rods, and bacteria united in pairs, and were usually non-motile. Occasionally a fungus was isolated which also formed manganese oxide freely. Fresh gravel, sand, calcium carbonate, and manganese oxide have the same property

of retaining manganous compounds from water. The removal of manganous compounds from water by filtration is effected by their retention by the filtering media, bacterial oxidation to manganese dioxide, retention of further quantities of manganous compounds by the newly formed manganese dioxide, and the oxidation of the retained matter by further bacterial action etc.

W. T. LOCKETT.

Removal of dissolved organic substances from water for drinking and general purposes. H. LÜHRIG (Gas- u. Wasserfach, 1927, **70**, 667—671, 684—688).—Sand from the filter bed for removing iron compounds from water becomes coated with a black deposit of manganese oxides. The fresh sand is capable of removing little manganese from river water containing manganese sulphate, but this power increases until with the "black sand" (containing about 2% Mn) the filtered water is completely free from manganese, and is greatly improved as regards colour and oxygen absorption value. The removal of organic substances by the manganese oxides is due essentially to adsorption, though the oxides may also function as oxygen carriers, and this efficiency is gradually reduced by the clogging action of the adsorbed substances. The efficiency can be restored by rubbing the sand or by washing it with water or with potassium permanganate. The de-manganising action of the sand increases with time, and both effects proceed independently. A synthetic "black sand," made by washing sand with a solution of manganese salts, is equally effective as that obtained from the filter bed.

W. T. K. BRAUNHOLTZ.

Chlorine absorption of water. J. R. MEADOW and H. HALE (J. Amer. Water Works Assoc., 1927, **18**, 75—81).—Ten-minute absorption tests, using 1·0, 3·0, 5·0, and 10·0 p.p.m. of free chlorine, showed that the amount of chlorine absorbed depends upon the amount of chlorine added, and extended tests on numerous samples of potable and non-potable waters using the 10-min. chlorine absorption method and the A.P.H. Standard (permanganate) method for oxygen consumed showed that, although in a great many instances the two methods were comparable, no strict correlation could be established between them. According to Keiser, for the determination of organic matter in water free from albuminous products the permanganate process (Kubel) and the determination of the chlorine number of Froboese are equally good, but the content of organic matter in water which has been polluted with sewage can be exactly fixed only by means of the chlorine absorption test. Experiments along similar lines with tap-water containing increasing amounts of urine showed that albuminous products, *e.g.*, urine, were not readily attacked by permanganate, but they absorbed substantial amounts of chlorine. According to all data, correlation between the two methods is good as long as no albuminous material is present. In the latter case the chlorine absorption test is the only safe criterion.

W. T. LOCKETT.

Cement-lined water mains. H. Y. CARSON (Ind. Eng. Chem., 1927, **19**, 781—783).—Cement lining of iron pipes retards corrosion in a more positive manner than do other coatings. At first there is a diffusion of calcium hydroxide from the cement to the water, which gives a zone in which the p_H value is high enough to precipitate and flocculate any iron, so that a thin membrane of iron rust is built up in the cement, thus retarding the action of the water on the pipe and on the cement. A thin coating of cement on iron exposed to atmospheric conditions is not so efficient as when the iron is under water, owing to the different conditions; in air the cement coating is flaked off by expansion of the products of corrosion whereas in water these are washed away. The former method of coating pipes with cement when set vertically, the cement being poured between a cylinder inside the pipe and the pipe wall, is superseded, a denser and smoother coating being formed by distributing the cement in a pipe resting horizontally upon roller bearings and rotating the pipe.

W. G. CAREY.

Base exchange in permutites. (Cationic exchange in eugels.) G. WIEGNER and H. JENNY (Kolloid-Z., 1927, **42**, 268—272; cf. Jenny, A., 1927, 415).—The relation between the power of exchange of an ion and the electric charge and degree of hydration is discussed. The exchange constants are directly proportional to the true ionic volume. With increasing dehydration, the exchange from liquid to solid phase increases. The exchange from solid to liquid phase depends upon the valency of the cation, on the degree of hydration, and on the solubility of the compounds possible in the permutite. These factors are considered.

L. L. BIRCUMSHAW.

PATENTS.

Preparation of a water-softening [base-exchange] reagent. A. S. BEHRMAN, Assr. to GENERAL ZEOLITE Co. (U.S.P. 1,624,711, 12.4.27. Appl., 1.10.21).—Greensand is treated with 2% hydrochloric acid, washed, treated with slightly alkaline sodium chloride solution, and, finally, with boiling sodium silicate solution,

T. S. WHEELER.

Water still. E. V. OLIVER (U.S.P. 1,629,807, 24.5.27. Appl., 10.5.20. Renewed 6.10.24).—The water is distilled from a well in the lower end of a receptacle provided with a hollow condenser within its upper portion. A trough, extending around the inner side of the wall of the receptacle and spaced above the well, receives the distillate from the condenser and defines a space for the upward passage of the steam from the well. Water is supplied to the lower end of the condenser. A tube extends upwards through the condenser and is provided near its upper end with an inlet for the passage through the tube of water from the upper end of the condenser. The lower end of the tube, discharging into the well, is positioned above the space defined by the trough. A rotatable cap is provided for adjustment of the inlet to the tube.

H. HOLMES.

Two-flow base-exchange water softener. C. T. MCGILL, Assr. to REITER Co. (U.S.P. 1,624,614, 12.4.27. Appl., 14.11.25).

Water softener. V. L. TANNEHILL, Assr. to FORT WAYNE ENGINEERING AND MANUF. Co. (U.S.P. 1,626,055, 26.4.27. Appl., 9.8.26).

Permanently preserving animals and plants. F. HOCHSTETTER and G. SCHMEIDEL (E.P. 274,314, 4.10.26).—See U.S.P. 1,602,489; B, 1927 30.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPTEMBER 23, 1927.

I.—GENERAL; PLANT; MACHINERY.

PATENTS.

Rotary drying apparatus. J. B. VERNAY (E.P. 262,464, 3.12.26. Conv., 5.12.25).—The apparatus comprises a number of concentric cylinders which are rotated, and through the spaces between which the material passes in zig-zag fashion from the centre outwards. Heated air may be introduced through a hollow shaft, and the material is fed through an opening surrounding the shaft by means of a conveyor device which also divides it into small pieces. If desired, the apparatus may be surrounded by a fixed casing in which a vacuum is maintained. B. M. VENABLES.

Drying apparatus. C. J. HUNT, Assr. to E. G. INFIELD (U.S.P. 1,623,540, 5.4.27. Appl., 20.11.26).—A structure contains a tumbling compartment round which air is circulated by being drawn from the sides of the structure and returned to the top. Air is also withdrawn from the lower part of the tumbling compartment. B. M. VENABLES.

Method of drying materials. J. PASQUIERS (E.P. 263,838, 28.12.26. Conv., 28.12.25).—The material is charged into a vessel which has a stirring device near the bottom arranged to give a slight lifting effect. Air for drying is blown through the porous bottom of the vessel, up through the agitated material. The air may be heated by external means or by the "heat of compression" and/or heat may be applied to the vessel.

B. M. VENABLES.

Drying and storing dried material preparatory to processing. W. F. HOSFORD, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,625,468, 19.4.27. Appl., 26.7.23).—The material is dried in one enclosure and transferred to another enclosure for storage, being maintained all the time independent of atmospheric conditions.

B. M. VENABLES.

Drying, heating, or the like apparatus. T. E. WOOD and H. HEYMANN (E.P. 269,064, 4.10.26).—In a dryer of many compartments, suitable for textiles, all the air is delivered by one fan, the delivery of which is divided up into a number of concentric pipes forming annular passages in which steam (for heating) and air alternate. Each chamber is supplied from one air annulus, and provision is made for supplying only new air to each compartment or for partial circulation of old.

B. M. VENABLES.

Apparatus for recovering solids from press liquids. H. E. BERGEN (U.S.P. 1,624,385, 12.4.27. Appl., 2.11.25).—The liquid is settled in a vertical cylindrical vessel which has a worm or other conveyor extending right across the bottom and out through a tubular member leading to a gate valve. Beyond the gate is another independently driven conveyor surrounded by a screen. B. M. VENABLES.

Cleaning filtering medium employed in the separation of mixtures of liquids, *e.g.*, oil and water, or mixtures of liquids and solids. H. M. ALEXANDER (E.P. 268,474, 20.1.26).—Non-inflammable liquids of low b.p. and sp. heat, having sp. gr. greater than that of water, *e.g.*, dichloroethylenes, trichloroethylene, carbon tetrachloride, tetrachloroethane, are forced in liquid or gaseous form through filtering or absorbent media to remove deposited oily matter, after as much as possible of the latter has been previously removed by a reverse flow of steam and/or air. The solvent is subsequently recovered by distillation. S. S. WOOLF.

Indicating the presence of submarine objects. C. W. BONNIKSEN and S. BARRATT (E.P. 268,004, 30.11.25, 1.12.25, and 28.9.26).—Flame and smoke are produced at the surface of the water by the ejection from a submerged object of activated phosphine contained in a cylinder. The activating agent is phosphorus dissolved in the phosphine or preferably a nitrogen compound containing oxygen, *e.g.*, nitric oxide, compressed in a similar cylinder and released simultaneously so that the two materials mix as gases. Suitable apparatus and releasing mechanism are described. W. G. CAREY.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Growth of mould fungi in coal. F. FISCHER and W. FUCHS (Brennstoff-Chem., 1927, **8**, 231—233).—Moulds have been observed to grow on raw lignite (not *in situ*), both before and after electrodialysis and with and without the addition of a nutrient medium. Growth occurs also in lignite that has been extracted with boiling water, and is especially vigorous on lignite after extraction with benzene and alcohol. W. T. K. BRAUNHOLTZ.

Determination of sulphur in coal. T. KIDOKORO and Y. KAMIGUCHI (J. Fuel Soc. Japan, 1927, **6**, 47—50).—The Probst method (B., 1924, 43) gives higher values than the Eschka method for the sulphur content of Japanese coals, the difference being approximately proportional to the organic sulphur content (difference $\div$ organic sulphur $= 0 \cdot 242$). The error of the Eschka method increases rapidly when the organic sulphur content exceeds 2%. Experiments with semi-cokes indicate that the error is closely associated with the content of non-volatile organic sulphur, and may be greatly diminished if care be taken to oxidise this completely. By the addition of sodium peroxide to the Eschka mixture (up to 2 g. per g. of coal) and subsequently heating for at least 2 hrs. at 800° accurate results are obtained. A. B. MANNING.

Improving the quality of Upper Silesian coke. G. DÖRFLINGER (Stahl u. Eisen, 1927, 47, 1166—1167).—The coal of the Pochhammer seam in the eastern coalfields of Upper Silesia is very rich in gas, but has only a poor coking power. A good quality coke can, however, be made from it by the use of narrow retorts and a high temperature if it is previously mixed with about 15% of a bituminous shale or of a fat coal from certain Lower Silesian mines. To test a series of coals for coking power each variety is first separated into the different coal constituents by the float-and-sink method using chloroform–xylene mixtures of different densities; the coking power and percentage of all the fractions are then determined, and from these figures a suitable mixture for feeding to the retorts is readily ascertained.

A. R. POWELL.

Equilibrium for the reaction $2CO = CO_2 + C$. A. STANSFIELD (Trans. Amer. Electrochem. Soc., 1927, **51**, 423—428).—The results obtained by Rhead and Wheeler (B., 1911, 129) for the equilibrium represented by the above equation are shown to be in error below 850°. The values of Boudouard (A., 1901, ii, 646) are in agreement with the calculated values from 450° to 800°.

L. M. CLARK.

Constituents of wood spirit and acetone oils. IV. H. PRINGSHEIM and A. SCHREIBER [with A. BEISER, W. DOSTER, H. LOOFMANN, G. POJARLIEFF, B. ROSEN, and E. STEINITZ] (Cellulosechem., 1927, **8**, 45—66. Cf. Pringsheim and Gorgas, A., 1924, i, 1158; Pringsheim and Bondi, A., 1925, i, 1072).—From the water-soluble portion (15%) of a sample of light oil from wood spirit, b.p. 80—125°, glycol dimethyl ether, allyl alcohol, and crotonyl alcohol were obtained. Glycol dimethyl ether was also present, together with trimethylacetaldehyde, methyl ethyl ketone, methyl propyl ketone, *iso*valeraldehyde, and *cyclo*pentanone, in the water-soluble fraction (35%) of the heavy oil. The water-insoluble fraction of the heavy oil, b.p. 135—150°, was treated with 50% sodium bisulphite solution, the insoluble portion (53%) was distilled with steam in the presence of dilute sulphuric acid to hydrolyse any esters, and then treated with sodium hydroxide solution to remove acids. On fractional distillation, the product yielded 2-methyl*cyclo*pentanone (b.p. 36—38°/11 mm., n_D^{20} 1·4431) and a fraction, b.p. 49—51°/12 mm., containing an unidentified *dimethyl*cyclo*pentanone* (n_D^{20} 1·4500; *semicarbazone*, m.p. 185°; *oxime*, m.p. 58°) and an oil having n_D^{20} 1·4733, which gave a pale red furan reaction, and did not form a semicarbazone. The dimethyl*cyclo*pentanone did not condense with cinnamaldehyde or *m*-nitrobenzaldehyde; it therefore contains at least one α-methyl group. The next fraction contained a ketone $C_8H_{14}O$, probably a *trimethyl*cyclo*pentanone* (b.p. 60—63°/12 mm.; n_D^{20} of freshly distilled sample, 1·4515, increasing after a few days to 1·4588; p-*nitrophenylhydrazone*, m.p. 155—156°; *oxime*, b.p. 115°/12 mm., n_D^{20} 1·4940). On oxidation with potassium permanganate and chromic acid the ketone yielded a small amount of *n*-butyric acid. The next fraction of the purified heavy oil consisted of 1:2:4-trimethylbenzene. A portion of the raw heavy oil, b.p. 131—137°, rapidly neutralised sodium hydroxide solution, and, on fractional distillation in a high vacuum of the acid oil obtained by acidifying the alkaline extract, *n*-valeric acid, which was present in the raw oil as the methyl ester, and small quantities of *n*-hexoic and *n*-heptoic acids, characterised as the silver salts, were isolated. The higher fractions yielded the lactones of three unknown acids, the *silver* salts of which were prepared and analysed: $C_9H_{18}O_4$, *lactone*, b.p. 97—100°/3 mm., n_D^{20} 1·4728; $C_{11}H_{20}O_4$, *lactone*, b.p. 99—100°/0·3 mm., n_D^{27} 1·4775; $C_{13}H_{22}O_4$, *lactone*, b.p. 113—115°/0·6 mm., n_D^{20} 1·4884. Another sample of the crude oil yielded an acid fraction, b.p. 143—150°/16 mm., which was neutralised with ammonia and extracted with ether. On addition of silver nitrate to the aqueous layer, silver γ-hydroxyvalerate was precipitated, indicating the presence of valerolactone. The ethereal solution afforded an unsaturated *lactone* $C_{12}H_{20}O_2$, b.p. 95—107°/0·3 mm., which, after prolonged boiling with dilute sodium hydroxide solution, was partly converted into the free *acid*, $C_{12}H_{22}O_3$, isolated as the *silver* salt. Propionic acid and an *acid* $C_9H_{16}O_4$ of unknown constitution were obtained from this lactone by oxidation with potassium permanganate; it may, therefore, be represented by $CH_2Me\cdot CH:C_9H_{14}O_2$. The corresponding *ketonic acid* $C_{12}H_{20}O_3$, b.p. 130°/14 mm., was also obtained by oxidation of the lactone. In another experiment 400 c.c. of acid oil obtained from 10 litres of crude heavy oil were carefully fractionated. The fraction of b.p. 202—205° contained Δ^{α}-*iso*hexenoic acid, the silver salt of which was prepared, and which was characterised by oxidation with potassium permanganate, which yielded *iso*butyric and oxalic acids. The fraction of b.p. 218—223° consisted of Δ^{δ}-heptenoic acid, characterised by means of the silver salt and by the formation of glutaric and acetic acids on oxidation. The higher fractions contained the lactone $C_{12}H_{20}O_2$ already mentioned, together with a *lactone* $C_{14}H_{24}O_2$, b.p. 120—126°/0·3 mm., which was not obtained in a pure state, but was converted into the *silver* salt of the corresponding acid, and a *lactone* $C_{17}H_{28}O_3$, b.p. 137—147°/0·3 mm., from which the *silver* salt was prepared. The quantities of these lactones obtained were insufficient for a determination of their constitutions. The light acetone oil used for this investigation had d^{21} 0·841, n_D^{21} 1·4029, and contained 13% of water. The dry oil had d^{21} 0·848; n_D^{22} 1·4030; 5 c.c. required 2·2 c.c. of 0·1*N*-sodium hydroxide solution to neutralise free acid, and 16·01 c.c. to hydrolyse the esters present. The oil was first freed from acids and esters by means of sodium hydroxide solution. The ketones were separated from traces of aldehydes by converting them into the bisulphite compounds and decomposing the latter in weakly alkaline solution. The product so obtained was then fractionally distilled. The following ketones were identified and characterised by means of the semicarbazone or *p*-nitrophenylhydrazone: acetone, methyl ethyl ketone, methyl *n*-propyl ketone (b.p. 101—103°), ethyl *iso*propyl ketone (b.p. 113—117°; the p-*nitrophenylhydrazone* has m.p. 84—86°, is unstable in air, and even in a vacuum desiccator rapidly becomes a dark brown viscous mass), and a small amount of ethyl *n*-propyl ketone (semicarbazone, m.p. 113°). A sample of heavy or "yellow" acetone oil was treated with sodium bisulphite solution, and the bisulphite compounds were decomposed with dilute acid.

On fractionation of the resulting oils, the following substances were isolated: 3(?)-methyl*cyclo*hexanone, b.p. 64—66°/15 mm., n_D^{20} 1·4395, 1 : 3(?)-dimethyl*cyclo*hexan-2-one, b.p. 70—75°/15 mm., n_D^{20} 1·4484, 1 : 1 : 3(?)-trimethyl*cyclo*hexan-4-one, b.p. 77—80°/15 mm., n_D^{20} 1·4595, dimethyl*cyclo*hexenone (unidentified), b.p. 83—85°/15 mm., n_D^{20} 1·4671, 1 : 1 : 2-trimethyl-Δ^2-*cyclo*hexen-4-one, b.p. 90—91°/15 mm., n_D^{20} 1·4827. The sodium bisulphite solution, after removal of the bisulphite compounds and unchanged oil, was made alkaline and steam-distilled under diminished pressure. On extraction of the distillate with ether and removal of the solvent, the extract was fractionally distilled. The following were isolated: heptaldehyde, characterised by preparation of the semicarbazone, m.p. 108—109°; an *aldehyde* of unknown constitution, $C_8H_{16}O$, b.p. 63—64°/18 mm., n_D^{27} 1·4362 (*semicarbazone*, m.p. 145—147°); *cyclo*hexanealdehyde, b.p. 76—77°/18 mm., n_D^{23} 1·4441 (semicarbazone, m.p. 164—166°), and β-*cyclo*hexylpropaldehyde, b.p. 87—89° /18 mm., $n_D^{24.5}$ 1·4630 (semicarbazone m.p. 132—134°).

W. J. Powell.

Jet and jetonised material. E. H. C. Craig (J. Inst. Petrol. Tech., 1927, **13**, 343—362).—Spanish jet showed a much flattened vegetable structure, and appeared to be derived from wood from a conifer. Whitby jet showed far less distinct vegetable structure. When jet was retorted in a Struben still, 3% distilled as water at 100°, 5% as water between 160° and 250°, 19% as light oil (*d* 0·792) below 300°, waxy oil at 300—320°; the total yield of oil and wax was 57·8 gals. per ton. 7·7% of powdered jet was soluble in kerosene. The examination of several lignites and a torbanite indicated that jetonised material is an intermediate stage between wood (or other vegetable matter) and coal or petroleum. Lignites are more closely related to coal than is jet. W. N. Hoyte.

Comparison of qualities of asphalts of German and Mexican origins. L. Rosner (Petroleum, 1927, **23**, 609—610).—German Elwerath crude oil and Panuco crude oil were distilled to asphalt in a pipe still without vacuum at 400—420°. The respective properties of the oils were: sulphur 0·55% and 5·8%; d^{15} 0·918 and 0·986; wax content, 1·2% and 0·2%, and asphaltum insoluble in benzine 0·1% and 16·4%. The asphalt from the Panuco crude is the more valuable, having higher drop point (Ubbelohde) and lower penetration and breaking points. The respective yields of asphalt were 8·00% and 50·60%. H. Moore.

Action of gases on glasses etc. Faber.—See VIII.

Oils in emulsions. Baldwin.—See X.

Patents.

Coke oven. T. G. Kus, Assr. to American Coke & Chemical Co. (U.S.P. 1,635,679, 12.7.27. Appl., 4.10.19).—A coke oven has regenerators arranged beneath and parallel to the coking chambers, flues connecting alternate regenerators with the bottoms of the combustion chambers, flues connecting intermediate regenerators with the tops of the combustion chambers, and burners in the tops and bottoms of the combustion chambers. A. B. Manning.

Coke oven. E. Coppée & Cie. (E.P. 253,887, 4.6.26, and 273,630, 2.5.27. Conv., 22.6.25 and 4.9.26).—The wall of the oven comprises an even number of groups of vertical flues, each of which is connected to its neighbour by a communication chamber. These chambers are arranged alternately at the top and bottom of the wall. The whole of the air for combustion is admitted in the outer group of flues, but the gas stream is divided, part entering each flue in the first group and part into the path of the burning gases through the ducts serving a group of flues. The means for admitting the gases to the upper communication chambers of two adjacent groups of flues is situated near to the dividing wall between the groups. At the base of the outer groups only, primary gas may be admitted through a duct short enough to preclude the formation of graphite.

R. A. A. Taylor.

Apparatus for coking coal. Urbana Coke Corp., Assees. of S. W. Parr and T. E. Layng (E.P. 256,192, 19.6.26. Conv., 30.7.25).—Coal is fed by screw conveyor through an inclined cylinder heated by waste gas or by gas or oil burners at a temperature below the plastic stage of the coal (cf. E.P. 249,886; B., 1927, 35), and thence into a heat-insulated hopper or direct into retorts of calorised iron or iron–chromium–nickel alloy. These are heated by gas or oil burners and the gases after combustion in outer vertical flues pass into direct contact with the retorts and thence to the coal preheater. The process is particularly applicable for the production of coke from non-coking coals. A. C. Monkhouse.

Fuels for use in internal-combustion engines and for other purposes. E. G. E. Meyer (E.P. 269,222, 3.12.25. Cf. E.P. 262,363; B., 1927, 134).—Liquid hydrocarbons of relatively high b.p. and substantially devoid of constituents boiling below 100°, and of which 80—100% distils over at temperatures up to 300°, are used mixed with a small quantity of volatile basic material (*e.g.*, ammonia) and ethyl ether.

R. A. A. Taylor.

Production of gas. O. U. Bean (E.P. 269,004, 14.6.26).—Steam is admitted to a chamber, and so heated that it is dissociated into its elements. A catalyst may be used to promote this reaction. A hydrocarbon vapour is then admixed with the hydrogen and oxygen, and the mixture is subjected to the influence of the elevated temperature, fluctuations in pressure, and, if necessary, of a catalyst. A gaseous fuel resembling natural gas results, together with the possible formation of light hydrocarbons and carbon black. Free oxygen appears in the gaseous product, and may be separated.

R. A. A. Taylor.

Low-temperature distillation of bituminous substances. Kohlenveredlung Ges.m.b.H. (E.P. 244,456, 5.12.25. Conv., 11.12.24).—A protective coating of aluminium or of an aluminium alloy lines the iron retort and prevents corrosion of it by the products of distillation. R. A. A. Taylor.

Distillation of coal at low temperatures. Comp. des Mines de Vicoigne, Nœux, et Drocourt (E.P. 255,411, 20.1.26. Conv., 17.7.25).—Fine coal and a tar or oil are mixed and moulded. They are then distilled in chambers by the passage over them of a hot

atmosphere of steam or other gas poor in oxygen, the temperature rising progressively to 500—600° in 3—12 hrs., when a good smokeless fuel results.

R. A. A. Taylor.

Manufacture of amorphous carbon etc. J. W. MacDonald, Assr. to W. L. Rucker (U.S.P. 1,626,418, 26.4.27. Appl., 14.12.25).—Ozokerite, paraffin, distillates, etc. are burnt by melting in a vessel which communicates by means of a valved pipe with a burner contained in a closed housing.

C. O. Harvey.

Polymerisation of oils. Barrett Co., Assees. of S. P. Miller (E.P. 246,491, 22.1.26. Conv., 23.1.25).—The cooled oil is introduced into a vessel in which it is agitated with sulphuric acid, aluminium chloride, or other polymerising agent, the requisite amount of which is added by means of an automatic device. After treatment the mixture is withdrawn, and the spent polymerising agent, together with any precipitated tar, is separated from the oil, which may then be washed and neutralised with alkali. The oil is treated in successive batches, all the operations, however, being automatically controlled so that in effect the process is continuous.

A. B. Manning.

Expelling sulphur dioxide gases from mixtures of sulphur dioxide gases and oil. Allgem. Ges. für Chem. Ind. m.b.H. (E.P. 269,118, 14.5.26. Conv., 12.4.26).—The mixture flows through a vertical pipe, externally heated by steam or hot air, and then passes into a superposed horizontal drum, so that the sulphur dioxide is driven off by the heated oil in the vertical pipe and is entirely separated from the oil in the drum.

W. G. Carey.

Apparatus for cooling coke. Humphreys & Glasgow, Ltd., Assees. of W. T. Bosler (E.P. 264,796, 2.11.26. Conv., 20.1.26).

Heating walls for coke ovens. Koksofenbau u. Gasverwertung A.-G. (E.P. 270,232, 25.8.26. Conv., 28.4.26).

Generating the supplementary steam for gas producers. Rasselsteiner Eisenwerks-Ges., A.-G., and A. Bien (E.P. 272,725, 9.9.26).

Gasification of solid fuels. L. Chavanne (E.P. 266,656, 10.2.26. Addn. to E.P. 247,571).

Compositions for roadways (E.P. 268,950).—See IX.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Calcium sulphate. III. New methods of analysis and use of plaster. L. Chassevent (Ann. Chim., 1927, [x], 7, 43—68).—A calorimetric method (cf. B., 1927, 676) for the analysis of plasters is developed, by which the course of the heating of the plaster may be followed, and a knowledge of its ultimate properties rapidly obtained. Anhydrous calcium sulphate, dehydrated below 300°, is rehydrated practically instantaneously, and a measurement of the heat evolved in g.-cal. during the first 2—3 min. (X) is due to this cause, so that the weight in grams (p) of anhydrous calcium sulphate in the sample is given by the expression $p = (X - 20)/20$, the heat of dissolution of the hemihydrate being 20 g.-cal. Calcium sulphate dehydrated between 300° and 600° requires 7 days for complete rehydration, and the period necessary for any particular sample may be determined either (1) by measuring the electrical resistance which, when the plaster contains no calcium sulphate dehydrated above 300°, does not increase after several hours; or (2) by a determination of the increase in weight of a known weight of the powder kept under water and dried at 50—60° after periods of 2, 7, and 15 days; all increase in weight between 2—7 days corresponds with the hydration of calcium sulphate which has been heated between 300° and 600°, and any increase after 7 days with that heated above 600°, 1 g. of anhydrous calcium sulphate requiring 0·26 g. of water for hydration. By this method the influence of the method of heating on various samples has been investigated. With samples prepared from precipitated calcium sulphate with insufficient heating a smooth temperature-time curve is obtained showing that hydration is continuous, but as the period of heating is increased a step curve results, the initial evolution of heat corresponding with the hydration of the anhydrous calcium sulphate to the hemihydrate, no further evolution of heat then occurring until crystallisation of the dihydrate sets in. With a plaster prepared from gypsum at 145° crystallisation sets in rapidly owing to the presence of seeds of the dihydrate being present, but removal of these by heating at 200° lengthens the period before crystallisation sets in. A good commercial plaster shows a steep step curve. A sample of dead plaster (which had been kept for 2 years) does not set owing to the formation of a skin of the dihydrate over each grain, which prevents diffusion of the supersaturated solution of the hemihydrate throughout the mass, each grain setting separately. The plaster may be restored by complete dehydration at 220°. The lack of hardness in set plaster has been investigated, and may be due either to the presence of overburnt particles which rehydrate only slowly, or to the addition of too much water, the best proportion being 45—60 g. per 100 g. of plaster. By mixing plaster with only slightly more than the theoretical quantity of water required for hydration at 80—90° and subjecting the mass to pressure (varying with the conditions and the size of the particles employed), samples having an apparent density greater than 2, a high resistance to compression, and an appearance like marble have been obtained. An attempt thus to prepare artificial alabaster was unsuccessful, the samples always being opaque and having a density less than 2·3. If plaster is hydrated with boiling water, care being taken that the temperature of the mass never falls below 63°, and the liquid mass is stored in steam-jacketed vessels, it remains plastic for many hours, but on allowing the temperature to fall to 40° it sets in the usual manner. By this means a smaller quantity of water is required for hydration, the plaster may be stored during use, and machinery employed to mix and work it.

J. W. Baker.

Patents.

Removal of cyanides from masses containing them. C. B. Jacobs, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,623,599, 5.4.27. Appl., 10.3.21).—The residue from the treatment of a mixture of alkaline

carbonate, carbon, and ferric oxide with nitrogen at high temperatures is treated at 40° with sufficient mother-liquor from a previous extraction to dissolve the alkali cyanide present. The extracting liquid is removed from the residue by centrifuging, followed by pressing at about 270 atm. T. S. Wheeler.

Removing deterrent salts from pickling liquors. G. S. Morgan (U.S.P. 1,626,623, 3.5.27. Appl., 30.6.26).—Sulphuric acid is added to the solution, to precipitate iron sulphate, until the acid content reaches 35—50% of the solution; the liquor is then drawn off and the precipitate dissolved in hot exhausted liquor and cooled to form copperas. H. Royal-Dawson.

Production of articles containing silicon carbide. Gebr. Siemens & Co. (G.P. 438,065, 13.5.24).—A plastic mass containing tragacanth, alone or mixed with water-glass, and powdered silicon carbide, with or without the addition of other material, such as mixtures of carbon with silica, silicon oxychloride, or silica, is moulded into the desired shape. L. A. Coles.

Preventing explosions in chambers used for grinding calcium carbide. Gräflich Schaffgotsch'sche Werke G.m.b.H. (G.P. 437,750, 18.9.25).—Combustion gases obtained by passing dry air through glowing coke are passed through the chambers at a temperature above 150°. L. A. Coles.

VIII.—GLASS; CERAMICS.

Testing glass vessels for holding drugs according to the specifications of the German Pharmacopœia, 6th ed. L. Kroeber (Apoth.-Ztg., 1926, **41**, 1406—1407; Chem. Zentr., 1927, 1, 785—786).—Glass vessels in which no formation of fine crystals takes place for at least ½ hr. on filling with freshly prepared and filtered 0·1% narcotine hydrochloride solution, are suitable for most purposes, whilst vessels in which no crystals appear after 3 days are suitable for the most stringent use. Medicine bottle glass should be suitable for general use if it does not discolour a test solution containing 3 c.c. of 0·1*N*-hydrochloric acid and 5 drops of methyl red per litre, on heating for ½ hr. on the water-bath. L. A. Coles.

Abstraction of sulphur dioxide from flame gases by glasses and glazes. A. Faber (Gas- u. Wasserfach, 1927, **70**, 419—422).—Summarising available evidence, it could be said that the surface action on glasses and glazes which occurred with gases containing sulphur and under fixed firing conditions, depended upon the concentration and the state of oxidation of the sulphur compounds. The composition of the glass was of importance, whilst the velocity of the gas and the temperature had been found to affect the results obtained. The conclusions of Travers on the action of sulphur trioxide on glass (B., 1921, 510 A) had been confirmed by Berdel, Ortmann, Zschimmer, and others. In technical furnaces the action of sulphur could be avoided by adjusting the firing and working conditions, *i.e.*, by a reducing atmosphere at first, followed by oxidising conditions. A. Cousen.

Influence of wet grog on the properties of saggars and other refractory goods. J. Konarzevski (Trans. Ceram. Soc., 1926, **25**, 386—393).—Two sets of grog fireclay bricks, one with dry grog and the other with wet, were prepared and fired in a commercial kiln to cone 10. Mechanical analysis of the grog showed it to contain 70% on the 8-in. sieve and 26% on the 20-in. sieve. Data concerning the contraction (wet to dry, dry to fired, wet to fired), sp. gr., porosity, tensile strength, cold crushing strength, and resistance to deformation at high temperatures, were obtained. The results show that the use of wet grog improves the properties of firebricks. The explanation appears to lie in the fact that clay sticks to wet grog much better than dry grog, and, in consequence, the particles of the brick are cemented more closely together during firing. A. T. Green.

Refractory linings for cement kilns. G. Martin (Trans. Ceram. Soc., 1926, **25**, 394—399).—A brief description of the process of burning cement in relation to the refractory linings of the rotary kiln is given. A cheap firebrick of fairly dense texture is satisfactory for the lining of the drying zone. The abrasive action experienced in the preheating zone makes a dense, non-porous firebrick essential for this portion. In the calcining zone the lining must be capable of withstanding the corroding action of lime at high temperatures. A satisfactory firebrick for this zone contains 62% SiO_2 and 32% Al_2O_3, a more highly siliceous product being undesirable. The brickwork in the clinkering zone is subjected to intense corrosion and abrasion by alkaline materials in continual movement, and to severe strains set up by the rotation of the kiln. There is diversity of opinion concerning the class of product for this zone. Alternate layers of high and low alumina brick have been used; the more siliceous bricks, being more deeply corroded, serve as holders for the attached cement clinker. Bauxite bricks, in a large-scale experiment, proved very satisfactory. In another instance, however, this material gave trouble through shrinkage. A kiln lined with firebricks, on which was placed a layer of material consisting of 2 pts. of small cement clinker and 1 pt. of cement, has given good service. A. T. Green.

IX.—BUILDING MATERIALS.

Calcium sulphate. Chassevent.—See VII.

Linings for cement kilns. Martin.—See VIII.

Patents.

Magnesite cement. J. Agerup (E.P. 268,989, 17.5.26).—Ferrous chloride is rendered anhydrous by drying the crystallised chloride or by rapid evaporation of a solution of the salt, and the ground product intimately mixed with magnesium oxide and suitable fillers, forming a magnesite cement which can be stored or transported without deterioration and used without skilled labour. B. W. Clarke.

Manufacture of slag cement. J. S. Harding, Assr. to W. R. Casparis and S. E. Stephenson (U.S.P. 1,627,237, 3.5.27. Appl., 13.1.23).—Lime is mixed with 70% or more of undried granulated slag and 3% or less of gypsum; after thoroughly drying the mixture, about 5% of cement clinker is added, and the whole is pulverised. H. Royal-Dawson.

Tar or tarry compositions applicable for roadways and the like. R. O. Child (E.P. 268,950, 23.3.26).—Coal tar or coal-tar pitch, dissolved in a suitable solvent, is agitated at 82° or less with partially coagulated casein, prepared according to E.P. 238,586 (B., 1925, 851) by treatment with sulphonated oil with or without the addition of formaldehyde, the casein being rendered homogenous by a preliminary treatment with a 5% Glauber's salt solution if necessary. B. W. Clarke.

Production of a plastic composition and articles therefrom. A. J. Slosser, Assr. to Pompeian Flooring Co. (U.S.P. 1,627,296, 3.5.27. Appl., 20.4.26).—A liquid binder solution of magnesium chloride is added slowly to a mixture containing approximately 45% by weight of vegetable fibrous material, 30% of asbestos fibre, and 25% of caustic calcined magnesia, producing a plastic mass which is spread or moulded into the desired form and allowed to set and harden.
H. Royal-Dawson.

Manufacture of plastic composition. J. F. Haggerty (E.P. 260,597, 18.9.26. Conv., 27.10.25).—Finely-divided fibrous material, *e.g.*, newspaper pulp, saturated with water, in the form of a flocculent mass in which the dry weight of the fibres is not more than 10% of the weight of water, is mixed with plaster of Paris, so that the quantity of dry fibrous material is approximately 5% of that of the plaster. The product may be moulded, and is lighter and tougher than ordinary gypsum products. B. W. Clarke.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Influence of boundary films on corrosive action. L. H. Callendar (Proc. Roy. Soc., 1927, **A 115**, 349—372; cf. B., 1926, 328).—The local corrosion of metals in normal waters is governed by boundary films of a solid, liquid, or gaseous nature present on the metal surface, and is liable to occur wherever a metal tends to form oxide films or scales of a higher potential than the metal itself. The location of the primary cathode and anode areas on the metal depends upon the distribution of these films, which are determined by the presence of foreign substances on the metal surface (dust, grease, moisture, etc.) and by irregularities in the surface itself. When the metal is immersed in an electrolyte, the oxide film is the primary cathode and metal passes into solution at unoxidised parts of the surface, but subsequently the original location of the cathode and anode areas may be altered by variations in the oxygen concentration, by the spreading of the products of corrosion over the metal surface, and by other factors. The rate of corrosion is indicated by the "boundary resistance," *i.e.*, the resistance in ohms of the boundary films between the electrodes and the electrolyte, obtained by subtracting the resistance of the electrolyte from the total internal resistance of the experimental corrosion cell. The electrical method used for measuring the changes in resistance and *E.M.F.* was similar to that previously described (*loc. cit.*), the corrosion cell consisting of an aluminium anode and a platinum or oxidised aluminium cathode with various dilute salt solutions as electrolytes. The normal cathodic oxide film formed in air is so thin that it has little effect on the rate of corrosion, but the thicker films formed by heating give a high boundary resistance and must tend to reduce the rate of corrosion. Oxidising electrolytes also retard corrosion by increasing boundary resistance. With aluminium, the boundary resistance increases both with the thickness of the film and with the increasing dilution of the electrolyte. L. L. Bircumshaw.

Comparison of the alloying elements chromium, nickel, molybdenum, and vanadium in structural steels. H. J. French (Trans. Amer. Soc. Steel Treat., 1927, **11**, 845—884).—The low-alloy structural steels are shown to be interchangeable to a large degree, and the relation fatigue limit–tensile strength for the various steels may be made by suitable treatment to fall on the same curve. Molybdenum produces uniform hardness over large sections. The load-carrying capacity at high temperatures and resistance to chemical reagents of various high-chromium–nickel alloys are given. A chart shows the effect of chromium in decreasing the attack of hydrogen sulphide on iron; with increasing nickel the loss of weight rises. Under certain conditions nickel and chromium increase the resistance of steels to wear, and cast railway crossings are now being made of steel containing 0·50% C, 0·75% Cr, and 2·75% Ni. T. H. Burnham.

Tensile properties of boiler plates at temperatures between 20° and 600°. G. Urbanczyk (Stahl u. Eisen, 1297, **47**, 1128—1135).—The mechanical properties between 20° and 600° of four typical boiler plate steels have been determined after annealing for 1 hr. at 920° and cooling slowly. The elastic limit remains constant up to 220° and then falls slowly with rise of temperature, the tensile strength increases slowly to a maximum at about 275° and then falls rapidly, and the elongation decreases slowly to a minimum at 250—275°, then increases slowly. The impact strength reaches its maximum at 120° and a minimum at 500—600°. Tables showing the composition and variation of the mechanical properties with the temperature are given together with graphs for all the steels tested. A. R. Powell.

X-Ray studies on the decomposition of austenite. O. E. Harder and R. L. Dodwell (Trans. Amer. Soc. Steel Treat., 1927, **11**, 975—985).—*X*-Ray analysis of powdered austenitic steels showed that the austenitic structure was partially destroyed by filing. The presence of undissolved carbides was objectionable as they gave a large number of lines interfering with the α- and γ-iron lines. Although micro-examination indicated a definite recrystallisation from γ- to α-iron after 70 hrs.' immersion in liquid air, no increase in intensity of the α-pattern was found. It is suggested that the α-crystal nuclei do not grow to sufficient size to give an *X*-ray diffraction pattern. *X*-Ray and micro-examinations also gave discordant results on the effect of stress on austenitic structure. When the α-pattern was present in quenched steels its intensity increased on tempering at 100°. The γ-pattern completely disappeared on tempering at 190° except in manganese and high-nickel steels. The γ-lattice contracted on immersion in liquid oxygen and the α-lattice increased after tempering at 190°, so that there was a hysteresis effect in change of para-

meter with change of temperature. It is unlikely that cementite is dissolved as a unit in austenite or martensite. Density determinations from X-ray data indicate that carbon atoms cannot replace iron atoms in the γ-lattice but must occupy positions inside it. T. H. Burnham.

Growth of cast iron. W. Schwinning and H. Flössner (Stahl u. Eisen, 1927, **47**, 1075—1079).—Grey cast iron begins to grow after heating at 450°, and the growth is much more marked at 550°. Repeated heating at 550° for periods of 3 hrs. followed by cooling results in a slow permanent growth at first, accompanied by a gradual change in the microstructure characterised by an intergrowth of the pearlite and cementite regions and a slow decomposition of the cementite lamellæ along their edges. After a certain number of heatings (10—20), depending on the composition of the iron, rapid decomposition of the cementite occurs accompanied by a marked increase in the volume of the metal and in the number and size of the graphite inclusions. At 650° this decomposition is extremely rapid, and takes place during the first 2—3 heatings. A. R. Powell.

Light aluminium alloys. L. Losana and E. Frova (Giorn. Chim. Ind. Appl., 1927, **9**, 111—115).—Various ternary and quaternary systems containing aluminium as principal component have been studied. Aluminium alloys rich in silicon and containing also copper give fused parts of good mechanical properties. That with 3% Cu and 2% Si exhibits a tensile strength of about 17 kg./mm.^{2} and an elongation of 6%; these results are superior to those given by the aluminium–copper alloy with 6% Cu, the latter being also the heavier. Aluminium alloys rich in silicon and containing also zinc or tin have low breaking strengths and are, in many cases, brittle. Silicon-rich aluminium alloys containing magnesium have very low densities and excellent tensile strengths, but their elongations are poor. With aluminium–copper–magnesium–silicon alloys a tensile strength of 20 (17—18) kg./mm.^{2} and an elongation of 3—3·5 (about 8)% are obtainable. To binary aluminium–copper alloys not more than 2% of zinc can be added without reducing the elongation excessively, whereas addition of tin lowers the breaking stress without raising the elongation markedly, and introduction of magnesium to the extent of 2% increases the tensile strength, but lessens the elongation somewhat. Hence, addition of zinc or tin to the ordinary aluminium–copper alloys containing 6% Cu is of no great practical advantage. T. H. Pope.

Analysis of antimony alloys. H. Vignal (Ann. Chim. anal., 1927, [ii], 9, 193—196).—For the determination of metals other than tin 1 g. of alloy is digested with gentle heat with 40 c.c. of 50% tartaric acid and 15 c.c. of nitric acid, and 10 c.c. of 50% sulphuric acid and about 15 c.c. of water are added to the clear mixture. After 12 hrs. the liquid is filtered and the lead salt weighed. Copper is then determined by adding caustic soda in excess, boiling, adding 0·5 g. of lactose in small portions, and boiling till the precipitate of cuprous oxide is well formed. To determine iron and zinc the filtrate is treated with hydrogen sulphide. The precipitate, now free from alkali salts, is added to the copper precipitate, calcined, dissolved in nitric acid, and the solution diluted and electrolysed, when any remaining lead is practically entirely deposited. 10 c.c. of 50% sulphuric acid are then added and electrolysis is continued until the copper is completely deposited. Iron and zinc left in solution are determined by the usual methods. The alkaline filtrate containing the tin and antimony is neutralised till the precipitated sulphates are only redissolved with difficulty, 30 g. of oxalic acid are added, and the mixture is boiled for 1—2 hrs. with hydrogen sulphide passing through. When precipitation appears complete a few drops of ammonium sulphide are added, the mixture is filtered hot, washed, the precipitate redissolved in hydrochloric acid, potassium chlorate added, the whole boiled, filtered, a few more crystals of chlorate are added, and boiling is continued till no odour of chlorine remains. After cooling to 25—30° and addition of 2—3 g. of potassium iodide, the solution is titrated with sodium thiosulphate (49·5 g. per litre); antimony sulphide is then precipitated. Antimony may be determined directly if lead and tin are present, or only lead, but not in the presence of copper. In the first case the alloy is dissolved in nitric acid, and after calcination the oxides are treated with melted potassium hydroxide in a nickel crucible, redissolved in hydrochloric acid, and the solution is boiled and treated as above. In the absence of tin, antimony oxide is not completely insoluble in nitric acid, so that 0·1—0·2 g. of pure tin should be added. For the determination of tin when over 50% is present, dissolution is effected as for an alloy of lead, tin, and antimony, and the hydrochloric acid solution of oxides, with 1 g. of iron wire, boiled under a reflux condenser until the iron is completely dissolved, antimony precipitated, and tin in the stannous state. 5—6 c.c. of antimony chloride are added (about 0·2 g. Sb) and more iron wire, and boiling is continued. After adding boiling water and filtering, the precipitate of antimony is rapidly washed, reheated, and the stannous chloride titrated in the boiling filtrate with 0·5N-potassium permanganate solution.

D. G. Hewer.

***X*-Ray investigation of the internal stress in metals [copper].** S. Sekito (Sci. Rep. Tôhoku Imp. Univ., 1927, **16**, 343—355).—Copper wire was cold-drawn, and the internal stress present then, and after annealing at different temperatures, was determined from the breadth of the lines in its X-ray spectrum. When reduced to 2% of its initial cross-section by drawing, the internal stress corresponds to a widening of 0·3% of the original lattice-parameter, the directions of greatest stress being [111] and [100]. The breadth of the spectral lines decreases as the annealing temperature is raised.

W. Hume-Rothery.

Determination of very low concentrations of volatile oils in emulsions. [Separation of minerals]. E. J. Baldwin (Ind. Eng. Chem., 1927, **19**, 78—81).—Distilled water is introduced into a 500 c.c. separating funnel, and 10 g. of galena, sifted to 35—48-mesh, are introduced for each 100 c.c. of water taken. The desired amount of oil is added, and the whole shaken for 5 min. Sufficient of the emulsion is blown off into a flask to leave 5—10 c.c. of emulsion with the ore in the funnel. 15 c.c. of purified ether are then introduced into the funnel, which is stoppered and shaken. The separated ether layer is blown into a distilling tube, another

10 c.c. of ether are added to the funnel, and the operation is repeated. Four more portions of 10 c.c. of ether are subsequently added, in each case the ether layer being added to the distilling tube as the ether is distilled off. Distillation is effected by immersing the tube in water at 40°, and is continued until the residue just fills a 2 c.c. bulb fused on to the bottom of the distilling tube. It is next transferred to a 10 c.c container, which is then filled with alcohol, mixed, and placed in a stoppered Erlenmeyer flask. The oil in the original emulsion is determined by comparing the turbidity produced by 5 c.c. of the alcohol–ether solution in 100 c.c. of gelatin solution with that produced by a standard alcohol–ether solution. The gelatin solution contains 1 g. of gelatin and 5 c.c. of glacial acetic acid in 2 litres of water. The readings are made in the dark with a Kober nephelometer–colorimeter. The oil remaining in the emulsion after ore treatment is determined in a similar manner, and the oil adsorbed by the galena calculated by difference. The determinations were concordant within 1%. The method might be used for determination of fats in food products, with the substitution of acetone for alcohol in some cases. H. MOORE.

Relation between temperature and Hall effect in some alloys. J. OBATA (Japan. J. Phys., 1927, 4, 43—47).—The Hall effect in fields from 0—20 kilogauss has been determined at room temperature and at the temperature of liquid air for two samples of manganin (Cu 85·40, Mn 10·03, Ni 4·11, Fe 0·42; and Cu 80·88, Mn 12·72, Ni 4·88, Fe 1·27); an alloy Cu 87·35, Ni 9·21, Al 2·80, Fe 0·50; constantan (Cu 59·62, Ni 37·77, Mn 1·39, Fe 0·65); copper (99·52%); phosphor bronze; gold (99·12%); and nickel. The corresponding values of the Hall constant excluding gold and nickel are, respectively, —0·00055, —0·00052, —0·00032, —0·00116, —0·00046, —0·00051. Nickel and the above alloys (except phosphor bronze, in which the constant remains unchanged) are characterised by a positive temperature coefficient of the Hall effect, whilst gold and copper have a small negative coefficient. R. W. LUNT.

PATENTS.

Metallurgical process. [Recovery of copper and the precious metals from concentrates.] W. E. GREENAWALT (U.S.P. 1,634,497, 5.7.27. Appl., 6.12.26).—Concentrates containing copper are roasted and leached to remove the greater part of the copper and the residue is smelted to obtain a copper matte containing the precious metals. This matte is roasted and leached to extract the remaining copper, and the residues are treated for the recovery of the precious metals. A. R. POWELL.

Treatment of [precious metal] ores for the removal of sulphur, tellurium, arsenic, and the like. A. J. SUMMERTON (Austral. P. 22,041, 2.3.25. Conv., 15.3.24).—The finely-ground ore is mixed with a dilute acid and the pulp is passed over an amalgamated copper plate above which is arranged a parallel sieve-like anode. By passing a current between the plate, as cathode, and the anode the nascent hydrogen liberated at the plate causes the arsenic, tellurium, and sulphur to be removed from the ore as hydrides which are oxidised by the nascent oxygen evolved at the anode while the precious metals adhere to the amalgamated plate. A. R. POWELL.

Alloy. G. O. SMITH, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,634,343, 5.7.27. Appl., 18.9.24).—An alloy which retains considerable resiliency at red heat and is suitable for high-temperature springs contains not more than 30% Cu, 60—95% of metals of the iron group, and 5—40% of metals of the tungsten group. C. A. KING.

Treatment of ores with chloride solutions. N. C. CHRISTENSEN (U.S.P. 1,621,969, 22.3.27. Appl., 18.5.26).—Ground lead ore is treated with hot sodium chloride solution containing ferric chloride, and, when chloridisation is complete, the pulp is cooled, neutralised with calcium carbonate, and filtered, the lead chloride formed being extracted from the residue with clean, hot sodium chloride solution, from which it is recovered by cooling. T. S. WHEELER.

Gas for cutting and welding purposes. J. R. ROSE, Assr. to CARBO-HYDROGEN CO. OF AMERICA (U.S.P. 1,628,066, 10.5.27. Appl., 3.8.18).—The gas to be used in conjunction with oxygen consists of a mixture of hydrogen with 20% by vol. of methane. F. G. CROSSE.

Soldering articles. E. THOMA (U.S.P. 1,630,771, 31.5.27. Appl., 17.7.24. Conv., 7.9.22).—A process for soldering articles formed of linked metal rings comprises cleaning the material, electrolytically depositing a suitable solder of low m.p. on it, coating with a flux and with graphite to prevent flowing of the molten solder, and heating to cause the solder to unite each link. For silver mesh a solder containing silver and cadmium is employed. T. S. WHEELER.

Centrifugal separator and amalgamator. W. C. COLEMAN (U.S.P. 1,632,950, 21.6.27. Appl., 6.9.24).—An outer rotor provided with a mercury chamber is divided by an inner rotor into two compartments communicating through a passage at the lower part of the outer rotor, the passage being constricted vertically to retard the flow of material through the mercury chamber. H. HOLMES.

Chromium-plated carbon article [mould]. F. M. BECKET, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,630,688, 31.5.27. Appl., 1.12.24).—Moulds or chills for use in casting metals consist of graphite electrolytically coated with chromium. T. S. WHEELER.

Manufacture of malleable cast iron. M. KUBO (U.S.P. 1,634,586, 5.7.27. Appl., 12.6.22).—See E.P. 202,464; B., 1923, 1027 A.

Beneficiation of [iron] ores. CROMWELL & MURRAY CO., Assees. of C. P. McCORMACK (E.P. 251,959, 19.4.26. Conv., 5.5.25).—See U.S.P. 1,575,852; B., 1926, 412.

Case-hardening of steel articles. F. PACHER (U.S.P. 1,635,384, 12.7.27. Appl., 21.11.25. Conv., 17.11.24).—See E.P. 242,978; B., 1927, 369.

XI.—ELECTROTECHNICS.

Patents.

Insulating liquid. C. J. Rodman, Assr. to Westinghouse Electric and Manuf. Co. (U.S.P. 1,622,809, 29.3.27. Appl., 25.10.21).—A mixture of a hydrocarbon insulating oil with a chlorinated methane derivative, *e.g.*, carbon tetrachloride, and a chlorinated benzene derivative, *e.g.*, hexachlorobenzene, is of value as an insulating oil for switches, and the like. It is practically non-inflammable, and gives little gas when arcing occurs. T. S. Wheeler.

Manufacture of high electrical resistances. J. Bradley, and Metropolitan-Vickers Electrical Co., Ltd. (E.P. 273,025, 30.3.26).—Synthetic or other resin, celluloid, shellac, pitch, etc. is dissolved in a volatile solvent, and the solution incorporated with a filler, *e.g.*, wood flour, chalk, or asbestos, and with finely-divided, electrically conducting material, *e.g.*, carbon, silicon. The solvent is evaporated and the mixture moulded to shape. J. S. G. Thomas.

Protective coating for electrical conductors. H. T. Tillquist (G.P. 438,472, 30.6.25).—Iron tubes etc. are protected by a coating of electrolytically-precipitated chromium, which is very resistant to the action of moisture and vapours. J. S. G. Thomas.

Incandescence electric lamp. J. A. Heany (U.S.P. 1,635,320, 12.7.27. Appl., 28.6.18).—Leading-in wires made of a copper-lead alloy containing 5—10% Pb are connected to the lamp filament positioned in a lead-glass stem in the lamp. J. S. G. Thomas.

Application of coatings to wires, cables, and the like, and composition therefor. A. E. P. Girard and M. J. P. Roumazeilles (E.P. 253,906, 14.6.26. Conv., 16.6.25).—See F.P. 611,899; B., 1927, 579.

Method of electrically testing [the dielectric strength of] insulating layers [on wires]. Internat. General Electric Co., Inc., Assees. of Allgem. Elektricitäts-Ges. (E.P. 260,997, 3.11.26. Conv., 3.11.25).

Electric cells. J. W. Carpenter, and Edison Swan Electric Co., Ltd. (E.P. 273,837, 14.4.26).

[Regulating position of electrodes in] electric furnaces. British Thomson-Houston Co., Ltd., Assees. of J. A. Seede (E.P. 263,792, 20.12.26. Conv., 24.12.25).

[Device for] electrode operation in electric furnaces. Internat. General Electric Co., Inc., Assees. of Allgem. Elektricitäts Ges. (E.P. 264,851, 20.1.27. Conv., 20.1.26. Addn. to E.P. 259,615).

XII.—FATS; OILS; WAXES.

Reactivity of chloramine (sodium salt of *p*-toluenesulphochloroamide) with fats. B. M. Margosches and M. Frischer (Chem.-Ztg., 1927, **51**, 519).—The behaviour of fats with chloramine under varying conditions was studied, the chloramine being determined in acid solution by adding potassium iodide and titrating the liberated iodine, and in alkaline bicarbonate solution with arsenious acid. The iodine value of a fat, calculated from the chloramine which reacted with it, varied with the nature and proportion of the solvents used for the chloramine and for the fat, with the acidity or alkalinity of the liquid, and with the time and temperature of mixing, but under certain conditions results were obtained in substantial agreement with those given by the Hübler method. F. R. Ennos.

Detergent action of soaps. P. H. Fall (J. Physical Chem., 1927, **31**, 801—849).—The relative detergent action of various concentrations of olive oil, palm oil, tallow, "Green Arrow," and silicated "Green Arrow" soaps has been determined at 40° and 70° by a method which measures the amount of finely-divided manganese dioxide suspended by a detergent solution in a given time. The optimum concentration for all five soaps, at both temperatures, lies in the range 0·2—0·4%, and the detergent values decrease with an increase in temperature. Olive oil soap solutions are less effective at the optimum than the remaining soaps, which resemble each other closely under the same conditions. The method, which is fully described and discussed, has also been applied to sodium oleate, sodium silicate solutions, and to sodium hydroxide and alkaline salts. The relative detergent values of soaps differ with different dirts, owing to specific adsorption. If the ratio of soap to dirt is less than 1 : 25 or 1 : 30, the dirt is not stabilised, and flakiness and floating occur. Solutions of sodium silicates are similar to soap in peptising solid dirts, the silicates with a high silica content being the most effective at concentrations greater than 0·15%. The optimum concentrations for all the silicates lie in the range 0·0125—0·05%, and the detergent powers, which decrease with a rise in temperature, are less than those of the soaps. Addition of sodium silicate to soap solutions the concentrations of which are below the optimum enhances the detergent power of the soap more than does the addition of sodium hydroxide, carbonate, or phosphate. These latter are also able to peptise certain samples of manganese dioxide, the optimum concentration lying in the range 0·012—0·025%. Change of temperature has an effect similar to that mentioned above. The lowering of surface tension is not a safe criterion for determining maximum detergent action. The methods previously used for measuring this property are summarised and that of McBain (A., 1924, ii, 155) and co-workers is adversely criticised. A new method utilising the action of salts on suspensions of clay in soap solutions was also unsatisfactory. A *résumé* of previous theories of detergent action is given. L. S. Theobald.

Utility of the Bertram method [for determination of the higher saturated fatty acids]. J. Van Loon, also S. H. Bertram (Chem. Weekblad, 1927, **24**, 319—320, 320; cf. B., 1927, 450).—(A) The procedure laid down by Bertram cannot lead to accurate results if fatty acids are present having more than 9 and less than 16 carbon atoms in the saturated chain, and unless the oxidation of the unsaturated acids present gives rise to no monocarboxylic acids between the acids $C_9H_{18}O_2$ and $C_{16}H_{32}O_2$. The oxidation of the unsaturated acids present in parsley oil, for example, gives rise to lauric acid, which is not separated by the method laid down by Bertram. (B) A reply to the above. Saturated acids containing between 9 and 16 carbon atoms in the chain are, of course, not separated. Unsaturated acids having

the double linking removed from the carboxyl group by 8 or more saturated carbon atoms do not occur in edible oils. S. I. LEVY.

Oils in emulsions. BALDWIN.—See X.

Rancidity in lard. HUSA and HUSA. **Whale oil in lard.** GRONOVER and BLECHSCHMIDT.—See XIX.

PATENTS.

Manufacture of soft soaps. L. G. LEFFER and N.V. INTERNAT. QEEP Co. (E.P. 268,980, 3.5.26).—Oils, fats, fatty acids, or resins are saponified under pressure with the optimum quantity of potassium hydroxide solution to produce soap without further treatment. During the saponification, ½—2% of aliphatic or aromatic hydrocarbons, and/or 6—15% of cruciferous plant oil, *e.g.*, rape seed oil, and/or 5—20% of oils or fats containing unsaponifiable matter, *e.g.*, sheanut butter, are added, in order to influence the setting power of the soap. Substantially neutral soaps stable to heat and cold within wide limits are obtained. S. S. WOOLF.

Production of a detergent paste. M. BRANDEGGER (G.P. 437,245, 12.2.24).—A paste suitable for removing tar, resin, and oil stains, etc. is prepared by mixing vegetable starch with an emulsion of an organic solvent with water, with or without the addition of a dissolving agent, and subsequently hydrolysing part of the starch. L. A. COLES.

Cod-liver oil (U.S.P. 1,629,074).—See XIX.

XVI.—AGRICULTURE.

Determination of the lime requirement of soils by means of the state of saturation with calcium. A. GEHRING, A. PEGGAU, and O. WEHRMANN (Z. Pflanz. Düng., 1927, **A8**, 321—331).—A number of details have been further studied in regard to the technique of the authors' method for ascertaining the lime requirement of soils by determination of the exchangeable calcium after treatment with excess of calcium hydroxide and conversion of the excess into carbonate by passing in carbon dioxide and boiling. The conclusion is reached that in general the method gives satisfactory results, but that some points in connexion with it are not yet clear, and that, with certain soils, errors are not excluded. The method, in its present form, does not seem to be applicable to humic soils. C. T. GIMINGHAM.

Determination of the reaction of soil. R. GANSSEN, H. PFEIFFER, A. LAAGE, H. HALLER, K. UTESCHER, and M. TRÉNEL (Z. Pflanz. Düng., 1927, **A8**, 332—344).—Investigation of a large number of soils has shown satisfactory correlation between the exchange acidity, the p_H value, and the molecular ratio of the total bases and of calcium oxide to 1 mol. Al_2O_3 combined with 3 mols. SiO_2. The molecular ratio of CaO to 1 mol. $Al_2O_3 + 3$ mols. SiO_2 in that part of the soil which is decomposed by boiling concentrated hydrochloric acid appears to be suitable for detecting a shortage of lime and for determining the reaction of a soil. The determination of silica is liable to introduce errors; the most suitable method is by extraction with 2% sodium hydroxide solution. C. T. GIMINGHAM.

Colloidal fraction of certain soils having restricted drainage. W. L. POWERS (Soil Sci., 1927, **23**, 487—491).—In experiments with the separated clay fraction of soils, the rapidity of flocculation and the volume of coagulum increased with the valency of the flocculating ion. Flocculation by acids and by calcium salts was more rapid when the suspension was previously made alkaline with ammonia. Improvement in the physical condition of certain soils following treatment with sulphur is attributed, not to the sulphuric acid produced, but to increased calcium brought into solution by the acid. Titration curves of each of the soils examined had similar characteristics. Cataphoresis trials showed that between p_H 10 and p_H 2·5 the colloid migrates as with a negative charge, and without any indication of an isoelectric point. A. G. POLLARD.

Measurement of "suction forces" in colloidal soils. F. HARDY (Soil Sci., 1927, **24**, 71—75).—The method of Joffre and McLean (B., 1925, 858) is adversely criticised in that it is affected by, but takes no account of, the rate of permeation of water through soil in the immediate neighbourhood of the instrument. A. G. POLLARD.

Replaceable bases in some Oregon soils. R. E. STEPHENSON (Soil Sci., 1927, **24**, 57—64).—Acid soils lose replaceable calcium by leaching following the exchange of hydrogen ions for calcium in the colloid complex. Similarly, alkali soils result from the exchange of sodium for calcium ions. Heavy soils have a greater base exchange capacity than light soils, but not necessarily a larger amount of replaceable bases. It is considered that the development of both acidity and alkalinity in soils may be considerably retarded and in some cases obviated by suitable soil management. A. G. POLLARD.

Possible rôle of iron-depositing bacteria in the formation of hard-pan. C. S. MUDGE (Soil Sci., 1927, **23**, 467—470).—From samples of hard-pan, bacteria were isolated which precipitated iron in pure culture solutions. The probability that such organisms are directly concerned with the formation of hard-pan is discussed. A. G. POLLARD.

Reaction and degree of saturation of soils. J. VON CSIKY (Z. Pflanz. Düng., 1927, **9 A**, 1—19).—Data are presented for the p_H in aqueous and potassium chloride solution suspensions, and for the exchangeable and hydrolytic acidity of certain Hungarian soils. Although, in some cases, a considerable degree of base unsaturation is shown, the p_H figures are considerably higher than those obtained under similar circumstances with soils of more humid countries such as Germany and Scandinavia. This is explained by the poverty of Hungarian soils in humus, and by the fact that the soils of the humid regions receive greater quantities of saline fertilisers. Whilst individual data do not give trustworthy information as to the lime status of soils, yet with closely comparable soils fertile soils show a higher p_H and less exchangeable and hydrolytic acidity than infertile soils. G. W. ROBINSON.

Comparison of different methods for the determination of the degree of saturation of soils. S. GERICKE (Z. Pflanz. Düng., 1927, **9 A**, 20—34).—The author has compared the baryta method of Hissink, the barium chloride method of Bobko and

Askinasi (B., 1926, 1025), and the lime-water method of Gehring (B., 1926, 207) for determining the base saturation capacity of soils. The baryta method gave the highest figures, followed by the lime-water method, whilst the barium chloride method gave the lowest figures. There is no evidence as to which method is the most trustworthy, but it is pointed out that Gehring's method is most comparable with practical procedure in the liming of soils. G. W. Robinson.

Pipette method for mechanical analysis of soils, its theoretical basis, and suggestions for a simplified apparatus. P. Köttgen (Z. Pflanz. Düng., 1927, **9 A**, 35—46).—A discussion of the pipette method, and of the methods to be used for the preliminary dispersion. A simple apparatus is described, consisting of a cylinder and pipette with bent orifice. The suspension is forced into the pipette by blowing air into the cylinder by means of a rubber bulb. G. W. Robinson.

Uniformity and utility of data in soil solution analyses. W. B. Bollen and R. E. Neidig (Soil Sci., 1927, **24**, 69—70).—A plea for the adoption of a standard expression of soil solution analysis on the basis of equivalent parts per million. A. G. Pollard.

Lysimeter studies. W. M. Higby (Soil Sci., 1927, **24**, 51—56).—Drainage waters from concrete lysimeter tanks were examined. Early portions of the percolate correspond approximately to soil solutions. Applications of lime and manure, alone and in combination, increased the leachable calcium, magnesium, nitrate, sulphate, and potassium in relative quantities of the above general order. The loss of nitrate was approximately doubled by this treatment. The amount of phosphate in the drainage water was small in all cases, but decreased with the lime and manure treatments. A. G. Pollard.

Effect of manuring with Chili saltpetre on the sugar beet. J. Souček and F. Kraus (Z. Zuckerind. Czechoslov., 1927, **51**, 417—421).—Sodium nitrate to the amount of 300 kg./hectare (2·4 cwt./acre) was applied in three different ways: (1) total amount at sowing; (2) half at sowing and half at singling as a top-dressing; and (3) all at singling, the crops being lifted at the end of September and later at the beginning of November. It was found that with early harvesting the roots were not ripe, and their sugar content was lower than that of the unmanured crop. Both for farmer and factory the most advantageous procedure is late harvesting with the divided addition. Later manuring (*e.g.*, all at singling) was regarded as unremunerative. J. P. Ogilvie.

Assimilation of phosphates measured by the bacterial fixation of nitrogen. G. Truffaut and N. Bezssonoff (Compt. rend., 1927, **185**, 85—86).—Cultures of nitrogen-fixing bacteria growing in a medium free from phosphates were supplied with various natural phosphates as their sole source of phosphorus, the amount of fixed nitrogen being determined after 26 days. It is thus shown that the biological inferiority of fluophosphates is due to their insolubility and not to any toxic action of the fluorine. After dissolution in sulphuric acid their metabolic value increases tenfold. E. A. Lunt.

Chemistry of basic slags. E. Vanstone (J. Agric. Sci., 1927, **17**, 143—152).—Two basic slags, one a low-soluble slag containing 18% of calcium phosphate, and the other a high-soluble slag containing 30% of calcium phosphate, were extracted for long periods with boiling 10% ammonium chloride solution. The low-grade slag lost 70% of its lime and very little phosphate, the residue containing about 40% of calcium phosphate. With the high-grade slag there was also a considerable loss of lime and, in addition, of phosphate. The residue after 96 hrs.' extraction of low-grade slag corresponded with the formula $3·3\ CaO,\ 1·0\ P_2O_5,\ 4·5\ SiO_2$: that from the high-grade slag corresponded with the formula $4·09\ CaO,\ 1·0\ O_2P_5,\ 1·3\ SiO_2$. It is concluded that a silicophosphate is present in the high-grade slag, but not in the low-grade slag. Attention is directed to the effect of dressings of slag on the exchangeable calcium of soils. The phosphate in fluor slags is probably present as fluorapatite. G. W. Robinson.

Determination of nitrates in plant materials. G. T. Pyne (J. Agric. Sci., 1927, **17**, 153—161).—Determinations of nitrate by distillation with magnesium oxide and Devarda alloy at 100° are untrustworthy in the presence of organic matter. Two methods are proposed for use with plant juices. In the first, the juice, cleared by addition of an equal volume of alcohol and subsequent filtration, is diluted and reduced with finely divided Devarda alloy in the presence of sufficient sodium hydroxide to give a 0·1*N*-solution. After reduction, excess of hydrochloric acid is added, and the ammonia is distilled off under reduced pressure with calcium oxide. In the second method, the cleared plant juice is treated with titanous hydroxide formed by adding titanous chloride to the juice to which sufficient alkali has previously been added. After reduction, ammonia is distilled off as above under reduced pressure at 40—50°. G. W. Robinson.

XVII.—SUGARS; STARCHES; GUMS.

Desiccation of sugar samples in tins. K. Šandera (Z. Zuckerind. Czechoslov., 1927, **51**, 309—313).—Tins with lever-top lids are prescribed for use by the Prague sugar sample distributing office, and have been found to render good service, meeting all requirements for storage and transport. Under ordinary conditions, with atmospheric moisture at 65% and temperature at 15—25°, the loss of weight is negligible, not exceeding 0·07% per month. J. P. Ogilvie.

Determination of sulphur dioxide in sugars. R. H. Hurst (Trop. Agric., 1927, 66).—Titration with iodine volumetrically in the usual way gives results much higher than reality, but the following procedure is sufficiently reliable for factory control purposes: 5 g. of the sugar are dissolved directly in an excess (say 20 c.c.) of standard iodine solution to which a few drops of hydrochloric or acetic acid have previously been added, the sulphite being thus converted quantitatively into sulphate, after which the excess iodine is titrated with sodium thiosulphate, the starch indicator being added as late as possible. Results obtained in this way agree very well with those found by the sulphide-stain procedure (cf. B., 1927, 312). J. P. Ogilvie.

Valuation of raw [beet] sugars in respect of their affinability. O. Spengler and C. Brendel (Z. Ver. deut. Zuckerind., 1927, 229—242).—A procedure is described for determining the relative value of raw beet sugars for refining by mashing with a sugar liquor saturated 3° below laboratory temperature and centrifuging, the amount of syrup and sugar obtained being noted for the calculation of the centrifuging power of the sample and the yield, respectively, and the sugar being graded according to its colour. The amount of fine grain in a raw sugar is no indication of its centrifuging power, which depends also on the size and regularity of the grain. J. P. Ogilvie.

Decolorisation of thin-juice [in beet sugar manufacture] by activated carbon. A. Linsbauer (Z. Zuckerind. Czechoslov., 1927, **51**, 483—490).—On the principle that the presence of sugar decreases the decolorising effect, the application of decolorising carbon to the juices rather than to the thick-juice (syrup) is advocated, it being claimed that only 0·03—0·04% of Carboraffin is required for thin-juice, whereas with evaporator thick-juice double this amount is necessary. J. P. Ogilvie.

Improvements in the desaccharification of [beet] molasses by the Steffen process. J. Bergé (Sucr. Belge, 1926, **46**, 1—10).—Difficulty experienced in washing the cake is now overcome by giving it a preliminary washing with water until it shows signs of cracking, and then pumping through a magma of re-precipitated, purified saccharate with which the washing is finished. Further, the amount of lime used has been reduced mainly by securing a more intimate contact between molasses solution and lime powder, the consumption now being less than 100% of the sugar. Only about 3% of sugar is lost in the final residual liquor. J. P. Ogilvie.

Peligot's saccharine [$C_6H_{10}O_5$] and its isolation from [beet] molasses. K. Vnuk (Z. Zuckerind. Czechoslov., 1927, **51**, 460—466, 467—477).—Glucosaccharine (the lactone of saccharic acid) was prepared by evaporating beet molasses to dryness with sodium sulphate, extracting the residue with acetone, dissolving out the acid, and converting it into its barium salt, this being purified by re-precipitation ; a preparation was also made by boiling a solution of invert sugar with lime, and purifying in a similar manner. In consequence of its high dextrorotatory power, saccharine disturbs the double polarisation method of determining sucrose, and its effect is not eliminated by either the ordinary Clerget process or the Andrlík–Staněk modification of making both observations in acid solution. J. P. Ogilvie.

100° point of the saccharimeter. O. Spengler, C. Brendel, and J. Schwirblianski (Z. Ver. deut. Zuckerind., 1927, 419—428).—Kraisy and Traegel (cf. B., 1924, 608) and others having found a normal solution of sucrose to produce values less than 100°, viz., 99·82—99·89°, the authors have tested Jäckel's suggested explanation of the adsorption of air on the surface of finely-divided sugar (cf. B., 1923, 671 a) by dissolving sugar in a flask connected with a very sensitive capillary gauge for indicating any minute increase in volume. On dissolving 17·35 g. of finely-powdered sugar in 18·5 c.c. of water, and taking into account the contraction arising on dissolution, an increase of volume of 5·76 c.c./100 g., equivalent to 7·0 mg., was found, an amount incapable of affecting the result. Results of other experiments are adduced to prove that this explanation of the error will not suffice. J. P. Ogilvie.

Sucrose crystallisation. I. A. Kukharenko and B. G. Savinov (Nauch. Zapiski [Russia], 1927, **4**, 139—142).—The formation of " crystallons " is explained according to Kukharenko's theory, whereby crystallisation will not take place unless centres of crystallisation are introduced. Chemical Abstracts.

Pectins and the mellowness of wines. Semichon.—See XIX.

XIX.—FOODS.

Detection of hardened whale oil in lard. A. Gronover and A. Blechschmidt (Z. Unters. Lebensm., 1927, **53**, 250—252).—A consignment of lard from a refinery had been rejected on the grounds of containing tallow or hardened fat by reason of the low difference of m.p. given by the Bömer test. Further samples of lard from the same shipment were taken from the refinery, and samples of the original material as received from America. The original material gave a normal Bömer value of 75·2°, but the refined material gave one value of 71·0°, which strongly suggested adulteration, and two values of 69·2° and 68·1°, which clearly indicated that hardened fat had been added at the refinery. It was subsequently admitted that during the summer hardened fat, and during the winter arachis oil, were added to give the lard a good consistency. The Bömer test is therefore valuable for the detection of both tallow and hardened whale oil in lard. H. J. Dowden.

Effect of benzoic and cinnamic acids on the rate of development of rancidity in lard. W. J. Husa and L. M. Husa (J. Amer. Pharm. Assoc., 1926, **15**, 1071—1074).—In an endeavour to discover the constituents of benzoin responsible for its effect in retarding the rancidity of lard, experiments have been conducted with benzoic and cinnamic acids. The addition of either acid in quantities up to 4% of the lard was not effective in retarding the development of rancidity as measured by the Kreis test. This test cannot be used for the detection of rancidity in benzoinated lard because Siam benzoin itself gives a pronounced red colour in the test. E. H. Sharples.

Determination of gelatin in ice cream. R. E. Remington and L. H. McRoberts (Ind. Eng. Chem., 1927, **19**, 267—269).—Deductions of the percentage of gelatin by determinations of its specific rotation and nitrogen content (Ferris, J. Dairy Sci., 1922, **5**, 555, and Smith, B., 1919, 228 a) can only be applied to inspection work if it is permissible to apply average values for rotation and nitrogen content to any grade of gelatin likely to be encountered. From analyses of a number of samples of ice cream gelatins, it appears that the figures 14·84% for nitrogen and 117 for specific rotation (corresponding with 17·53 and 139, respectively, on the dry ash-free basis) may be taken as average values with a maximum error of 3% of the amount of gelatin present. Suggested modifications for the Ferris

method are the use of a smaller sample, weight instead of volume dilution, control of the casein precipitation by means of hæmatoxylin indicator, the use of alum to ensure complete precipitation of the gelatin, and the correction of the nitrogen found for that from the milk which comes through into the filtrate. Ferris' original and the modified method are described in detail.

C. O. Harvey.

Micro-determination of caffeine in coffee. A. C. Röttinger (Z. Unters. Lebensm., 1927, **53**, 146—151).—5 g. of finely-ground coffee are introduced into a separating funnel, the open end of which is ground to fit into the neck of a flask containing 100 c.c. of chloroform. After uniting the vessels the contents are agitated for ½ hr., 20 c.c. of the solution are filtered through a pad of cotton wool into a specially designed pipette, which fits the stop-cock of the separator. The solution is transferred from the pipette into a bulb-shaped vessel which is heated on a water-bath and the chloroform removed by aspirating air. To the residue in the bulb are added a small piece of paraffin wax, a few c.c. of ether, and, after warming, a few c.c. of dilute hydrochloric acid, and the whole is thoroughly shaken. The ether is removed by a current of air, and the acid layer drawn off into a small separating funnel, leaving the paraffin wax adhering to the walls of the bulb. The residue is extracted in this way four times with 0·5% hydrochloric acid, the extracts being collected in the same funnel. The acid solution is then shaken thoroughly with four successive quantities of chloroform, the chloroform layer being drawn off through cotton wool into a distillation flask, from which the chloroform is subsequently evaporated. The crude caffeine which is left is treated with 2 c.c. of concentrated sulphuric acid, potassium sulphate, and copper sulphate, and decomposed by heating for ½ hr., after which a Kjeldahl distillation is performed. If 0·02*N*-sulphuric acid is used, 1 c.c. of acid corresponds to 0·0969% of caffeine. The apparatus described reduces the total experimental error to about 0·02%.

H. J. Dowden.

Physiology of apples. VI. Correlation in the individual apple between the mineral constituents and other properties. J. W. Brown (Ann. Bot., 1927, **41**, 127—137; cf. B., 1926, 605).—Analyses of 30 Bramley's Seedling apples from the same orchard showed that green apples contained less dry matter, total ash, and potassium, and more nitrogen than yellow, and red and yellow apples; the p_H of the juice of green applies was also appreciably lower. High values for the p_H of the juice were associated with high content of total ash and potassium. Partial correlations between some pairs of constituents were calculated, and significant values showing direct correlation between potassium and phosphates, phosphates and acidity, and phosphates and iron were obtained. Potassium and iron, and potassium and acidity were inversely correlated. No significant correlations between nitrogen and any of the mineral constituents were found.

C. T. Gimingham.

Pectins of grapes and the mellowness of wines. L. Semichon (Chem. et Ind., 1927, **17**, 25—32).—By the addition of alcohol to a wine acidified with 1% of hydrochloric acid, the pectins and gums may be precipitated in a fair state of purity, the former being separated from the latter by preparation of the pectate of calcium. Grape musts contain pectins but no gums. Wines may contain a mixture of the two, or pectins may be absent, according to the stage of fermentation reached. No relation has been found between the amount of pectins which disappear and the gums formed. The pectins consist of the methyl ester of pectic acid, combined with other organic compounds, and with phosphates of calcium, magnesium, and aluminium. On hydrolysis they yield pentoses. The gums are neutral, show no ester character, and on hydrolysis yield hexoses. The influence of the pectin content of the grape must on the mellowness of the wine is discussed.

F. R. Ennos.

Detection and determination of nitrogen-bearing chemicals added to animal or vegetable nitrogenous materials. H. C. Moore and R. White (Ind. Eng. Chem., 1927, **19**, 264—266).—Ammonium sulphate and cyanamide have been found in nitrogenous tankage, and urea might be added. The addition of sodium nitrate would not increase the nitrogen content as determined in the usual way. The silver nitrate test for cyanamide is unsatisfactory for testing tankage directly, but may be carried out on the portion which settles to the bottom of the container when the tankage is mixed with carbon tetrachloride. This portion contains the heavy particles, such as ammonium sulphate, carbamide, etc., and the further addition of an equal volume of turpentine will cause crystals of urea, if present, to appear in partial suspension. Modifications of the ordinary qualitative reactions in general use are recommended for testing tankage. Nitrate nitrogen is determined on 50 c.c. of a filtered 2% aqueous solution of the tankage by reduction and alkaline distillation, the ammonia derived from ammonium salts being separately determined and deducted from the figure obtained. Cyanamide and dicyanodiamide are precipitated by the addition of excess of silver nitrate solution to 50 c.c. of a filtered 2% aqueous solution of the tankage, followed by 20 c.c. of 10% potassium hydroxide solution. Nitrogen is determined in the brown precipitate by the Kjeldahl method. The residue obtained by filtration of a solution of 5 g. of tankage in 250 c.c. of anhydrous alcohol contains the ammonium sulphate, which is determined by extracting with water, bulking to 250 c.c., and distilling 50 c.c. with 150 c.c. of water and excess of magnesium oxide. Urea is determined in 50 c.c. of the original alcoholic solution by adding 50 c.c. of water, boiling off the alcohol, adding 100 c.c. of water, cooling, adding 0·25 g. of urease, and, in ½ hr., distilling after the addition of 1 g. of paraffin wax and 5 g. of heavy magnesium oxide. A correction for the solubility of ammonium nitrate in alcohol must be made if this salt is present, by performing a separate alkaline distillation. The results obtained are sufficiently accurate for most purposes. The presence of more than 0·5% of free ammonia in nitrogenous tankage points to the addition of ammonium salts.

C. O. Harvey.

Oils in emulsions. Baldwin.—See X.

PATENTS.

Preservation of fruit. O. H. HANSEN, Assr. to HANSEN CANNING MACHINERY CORP. (U.S.P. 1,625,207, 19.4.27. Appl., 7.5.24).—Fruit is temporarily sealed in cans, which are then exhausted and filled with steam. Sufficient syrup to cover the fruit is admitted and the submerged fruit completely sterilised, the cans being finally sealed. T. S. WHEELER.

Removal of residual poisons [lead arsenate] from fruit. E. M. SEARS (U.S.P. 1,624,074, 12.4.27. Appl., 21.10.26).—Fruit which has been sprayed with lead arsenate can be completely freed from it by immersion in a mixture of a mineral or vegetable oil and a fatty acid, *e.g.*, stearic acid, followed by washing in a dilute alkaline solution and in water. T. S. WHEELER.

Production of feed stuff. C. S. MINER and G. STEBRUP, Assrs. to QUAKER OATS CO. (U.S.P. 1,627,921, 10.5.27. Appl., 28.5.21. Renewed 23.3.27).—Roughage, such as seed hulls, straw, and other vegetable products with a high crude-fibre content, is mixed with not more than its own weight of water and with about 8% of lime, and heated at 150° under a steam pressure of about 80 lb./sq. in. for 3 hrs. After allowing the mass to cool without the escape of vapours, it is dried to a moisture content of approximately 12%, and ground. F. R. ENNOS.

Manufacture of bread, cakes, and other yeast-leavened baked foods. W. D. STEIN (E.P. 255,469, 14.7.26. Conv., 29.8.25).—The flour is mixed with a monosaccharide, *e.g.*, dextrose or lævulose, and sufficient lactic acid to promote the most favourable acidity (p_H 4·8) for the growth of yeast. The dough is then made and baked. F. R. ENNOS.

Preparation of bakery produce. C. SCHMITT (G.P. 435,973, 22.3.25).—Addition of fine meal, obtained from cottonseed meal by sifting, raises the albumin content of biscuits, bread, etc. S. S. WOOLF.

Manufacture of a highly-concentrated substance rich in antirachitic and antixerophilic vitamins from cod-liver oil. C. FUNK and H. E. DUBIN, Assrs. to H. A. METZ LABORATORIES, INC. (U.S.P. 1,629,074, 17.5.27. Appl., 28.6.23).—Cod-liver oil is heated under reflux with an equal weight of glacial acetic acid for 8 hrs., the acid being concentrated to one tenth its bulk and extracted with benzene. The benzene solution is washed with water and evaporated to dryness, and the residue is heated with alcoholic sodium hydroxide solution. The solution obtained is diluted with water and extracted with benzene. The residue from evaporation of the benzene contains the vitamins of cod-liver oil in a highly-concentrated form. A still more concentrated preparation can be isolated by treatment of this residue with digitonin in the usual way. Finally, 1 kg. of oil yields 0·2 g. of a crystalline product. T. S. WHEELER.

Manufacture of milk powder. J. M. SIERRA (U.S.P. 1,609,384, 7.12.26. Appl., 13.3.23. Conv., 27.3.22).—See E.P. 198,496; B., 1923, 738.

Manufacture of a flour improver. D. J. BLOCK, Assr. to W. D. STEIN (U.S.P. 1,626,910, 3.5.27. Appl., 29.8.25).—See E.P. 255,469; preceding.

Treatment of meal, flour, and other milling products with per-compounds. J. VAN LOON (E.P. 245,801, 11.1.26. Conv., 10.1.25).—See F.P. 609,057; B., 1927, 265.

Drying chambers for vegetables and like products. G. SCOTT AND SON (LONDON), LTD., and G. W. RILEY (E.P. 271,143, 16.2.26).

Preparation of infusions of coffee or the like. C. URTIS (E.P. 270,586, 3.12.26).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Separation of the most important opium alkaloids. F. CHEMNITIUS (Pharm. Zentr., 1927, **68**, 307—310).—A method for the separation and purification of morphine, codeine, narceine, narcotine, thebaine, papaverine, and meconic acid is described. E. H. SHARPLES.

Microchemistry of narceine. M. WAGENAAR (Pharm. Weekblad, 1927, **64**, 354—357).—The alkaloid is readily precipitated by addition of potassium nitrite (and other salts of weak acids) to solutions of its salts, in bundles of fine needles, of negative double refraction. The needles are coloured blue or brown by free iodine. The chloride, bromide, and thiocyanate are obtained by addition of the corresponding ammonium or potassium salts to a solution in hydrochloric acid. Addition of platinic chloride produces an amorphous precipitate which soon becomes crystalline, but the crystals are not highly characteristic. S. I. LEVY.

Poisoning action of mercury alkyls. A. KLAGES (Z. angew. Chem., **40**, 559—561).—The fungicidal and germicidal action of various mercury alkyl halides is tested on fungus spores and on wheat kernels. The poisonous effect increases in the order chloride $<$ bromide $<$ iodide. The effect with these alkyl halides is considerably greater than with the mercurous halides. L. M. CLARK.

Stability of physostigmine [eserine] solutions. J. C. KRANTZ, JUN., and F. J. STAMA (J. Amer. Pharm. Assoc., 1927, **16**, 412—414).—The red colour which develops in solutions of physostigmine sulphate and salicylate due to the formation by hydrolysis of eseroline, can be prevented for a considerable period by adjusting the hydrogen-ion concentration to p_H 2—3 by means of hydrochloric, boric, sulphuric, or salicylic acid. Water saturated with carbon dioxide, with an atmosphere of the gas above, is the best medium to use for preventing the appearance of this red colour. E. A. LUNT.

Production of essences. F. O. SCHOEPPE (Riechstoffind., 1926, 92; Chem. Zentr., 1926, II., 3119).—The use of alcohol to render essential oils soluble in water may be avoided by the following method. The oil is mixed with 15—30% of a sulpholeate (Turkey-red oil) containing about 50% of fat, and the mixture is dried with 2% of anhydrous potassium carbonate. No deterioration in quality occurs. E. H. SHARPLES.

Bergamot. F. LA FACE (Riechstoffind., 1926, 85—87; Chem. Zentr., 1926, II., 3119).—The following characteristics are given for bergamot oil: d^{15} 0·880—0·887, α_D + 8° to + 24°, linalyl acetate, 35—45%; saponif.

value, 1 — 3·5; distillation residue in water-bath, 4·5—6·5%; solubility in 90% alcohol, 1 in 0·5 vol.

E. H. Sharples.

[Constituents of] Port Orford cedar wood oil. F. H. Thurber and L. J. Roll (Ind. Eng. Chem., 1927, **19**, 739—742. Cf. Schorger; B., 1914, 886).—Fractional distillation of Port Orford cedar wood oil yielded 46% of *d*-α-pinene, b.p. 156—156·5°, d^{20} 0·8584, n_D^{20} 1·4608, $[\alpha]_D^{20}$ + 53·01°, characterised by the preparation of pinene hydrochloride, m.p. 126—127°, and α-pinonic acid, m.p. 68°. This sp. rotation is the highest recorded for *d*-α-pinene. The redistilled product gave no trace of the inactive nitrosochloride, the formation of which is characteristic of nearly all samples of *d*-α-pinene obtained from American turpentine oil, and is therefore regarded as a pure *d*-α-pinene isomeric with the ordinary form. Oxidation yielded no trace of pinonic acid, indicating that β-pinene is not a component of the oil. The fractions of b.p. 159—178° and 78—100°/14 mm. (3% of the original oil) contained a small proportion of *d*-limonene, b.p. 170—176° (tetrabromide, m.p. 124°). The next fraction, b.p. 100—122°/14 mm., consisted of *d*-borneol, m.p. 201°, $[\alpha]_D^{20}$ + 32·6°, which was isolated as the acid phthalate, m.p. 164°, and represented 13·8% of the original oil, and a liquid secondary *alcohol* (11·5%), of mol. wt. equal to that of borneol, b.p. 104—107°/13 mm. Saponification values before and after acetylation showed that 5·2% of the total secondary alcohol in this fraction was present in the oil as the acetate, and 20·1% as the free alcohol. The fraction of b.p. 122—145°/14 mm. consisted of *d*-cadinene (21%), b.p. 272—274°, d^{20} 0·9255, n_D^{20} 1·5065, $[\alpha]_D^{20}$ + 103·7°, from which the dihydrochloride, m.p. 118°, was prepared. The highest fraction, b.p. 145—180°/14 mm., $[\alpha]_D^{20}$ —10·54°, equivalent to 3·9% of the oil, contained *l*-cadinol (cf. Semmler and Jonas, A., 1915, i, 63).

W. J. Powell.

Temperature coefficients for specific gravities and rotations of essential oils. N. Schoorl (Pharm. Weekblad, 1927, **64**, 451—453).—The coefficient for the rotation may be taken as 0·0004, and for the specific gravity as 0·00075; these are approximate average values only, but may be employed to correct readings taken under ordinary conditions. S. I. Levy.

Glass vessels for drugs. Kroeber.—See VIII.

Oils in emulsions. Baldwin.—See X.

Caffeine in coffee. Rottinger.—See XIX.

Patents.

Production of aqueous solutions of free cyanamide. A. M. Muckenfuss, Assr. to Roessler & Hasslacher Chemical Co. (U.S.P. 1,622,731, 29.3.27. Appl., 14.8.24).—Calcium cyanamide is treated in presence of cold water with sulphur dioxide at such a rate that, while the liquid remains neutral, calcium sulphite is precipitated and free cyanamide is formed. By filtering at intervals and adding fresh calcium cyanamide and sulphur dioxide, solutions containing up to 13·5% of free cyanamide can be obtained.

T. S. Wheeler.

Process of esterification. U.S. Industrial Alcohol Co., Assees. of F. B. Arentz (E.P. 246,526, 26.1.26. Conv., 26.1.25).—A continuous esterification process and plant are described which are applicable in cases where the alcohol or ester or alcohol–ester mixture can form with water an azeotropic mixture, *e.g.*, the preparation of *n*- or *iso*-butyl acetate, amyl formate, acetate, or phthalate, *n*-butyl tartrate or phthalate. The mixture of acid and alcohol is heated under a reflux condenser which allows the vapours of the azeotropic mixture to pass forward to a descending condenser and a separator, from whence alcohol and/or ester is returned to the still and aqueous liquor is removed. If a water layer forms in the still it may be drawn off in order to economise time and fuel. The preparation of *n*-butyl acetate and tartrate is detailed. C. Hollins.

Manufacture of polyhydroxy-carbon compounds. H. Essex and A. L. Ward, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,626,398, 26.4.27. Appl., 25.5.26. Cf. U.S.P. 1,594,608; B., 1926, 897).—An olefinic compound, *e.g.*, ethylene or allyl alcohol, is treated with carbon dioxide in presence of sodium hypochlorite solution, and the resulting solution of the corresponding chlorohydrin and sodium carbonate is heated at the b.p. to form the corresponding polyhydroxy-compound.

T. S. Wheeler.

Manufacture of aliphatic keto- and aldehydo-carboxylic acids. C. H. Boehringer Sohn Chem. Fabr., Assees. of A. Häussler (U.S.P. 1,627,091, 3.5.27. Appl., 22.12.25. Conv., 5.3.23).—Esters of hydroxy-aliphatic acids, *e.g.*, ethyl lactate, are passed in the vapour phase with an excess of air, and preferably with steam, at 100—500° over oxygen compounds of metals capable of more than one degree of oxidation, *e.g.*, vanadium pentoxide, lead chromate. A 50—80% conversion into the corresponding keto- or aldehydo-ester, *e.g.*, ethyl pyruvate, is obtained. T. S. Wheeler.

Manufacture of glycols. Carbide & Carbon Chemicals Corp., Assees. of G. O. Curme, jun. (E.P. 264,124, 5.7.26. Conv., 7.1.26).—Chlorohydrins are converted into glycols by hydrolysis with a solution of caustic alkali under such conditions that any appreciable escape of the intermediate olefine oxide is prevented, *e.g.*, by using a vessel completely filled with liquid. The temperature and pressure are regulated so that the conversion of the chlorohydrin into olefine oxide and of the latter into glycol proceed at suitable rates.

B. Fullman.

Manufacture of [inactive] menthol. Rheinische Kampfer-Fabr. G.m.b.H., Assees. of K. Schöllkopf (U.S.P. 1,629,002, 17.5.27. Appl., 26.6.26. Conv., 11.3.22).—Thymol is treated with hydrogen at 200° and 5—30 atm., in presence of a nickel, platinum, or palladium catalyst, and the *r*-menthol formed is separated from liquid isomerides by freezing or fractional distillation. These isomerides are heated with sodium at 300° for 20 hrs., and the liquid product is distilled with steam and then fractionated. About 50% is obtained as *r*-menthol. The remainder is mixed with a fresh batch of thymol.

T. S. Wheeler.

Cyclic production of soluble salts of organic compounds of an acid character. M. Buchner (E.P. 246,142, 12.1.26. Conv., 16.1.25).—Acid organic

compounds, especially fatty acids or their esters (glycerides etc.), are treated with the hydroxides or carbonates of calcium, magnesium, etc., producing insoluble salts which are decomposed by a fluoride (simple or complex) to give the required soluble salt of the acid compound together with a fluorine derivative of the original metal (often insoluble). From the latter is reproduced the fluorine compound used in the reaction, *viâ* the complex fluoride. The metal compounds required for the production of the insoluble derivatives of the initial organic substance may be used cyclically; *e.g.*, if calcium carbonate be used, the calcium chloride obtained as above is reconverted into carbonate by means of the carbon dioxide liberated and ammonia. B. Fullman.

Compositions containing organic mercury compounds. E. C. R. Marks. From E. I. Du Pont de Nemours & Co. (E.P. 269,243, 12.1.26).—A mercury oxide or salt is caused to react with an organic compound (such as a phenol, negatively-substituted phenol, or naphthol, etc.) in the presence of a finely-divided solid material which acts as a diluent or vehicle for the organo-mercury compound formed, which is deposited on its surface. The diluent substance may be either inert (clay, charcoal, barium sulphate, etc.) or such that it reacts with one of the other substances present (hydrated lime, magnesium oxide or carbonate, etc.). *E.g.*, 30 pts. of *o*-chlorophenol are heated with the product formed by the action of 50 pts. of mercuric acetate on 500 pts. of hydrated lime in 2000 pts. of water. The final product contains 6·7% of the calcium salt of hydroxymercuri-*o*-chlorophenol. These substances have application as fungicides etc.
B. Fullman.

Production of 3-bromo-2-hydroxypyridine-5-arsinic acid. A. Binz and C. Räth (E.P. 263,142, 13.12.26. Conv., 21.12.25).—Bromination of 2-hydroxypyridine-5-arsinic acid (E.P. 250,287; B., 1926, 512) in acetic acid yields 3-*bromo*-2-*hydroxypyridine*-5-*arsinic acid*, which has trypanocidal and spirillocidal properties.
C. Hollins.

Manufacture of complex antimony compounds. H. Hahl, Assr. to Winthrop Chemical Co., Inc. (U.S.P. 1,628,838, 17.5.27. Appl., 29.3.26. Conv., 6.6.25).—Antimonyl compounds of pyrocatechol and its derivatives are heated in presence of water with sodium malonate or with a neutral salt of a similar carboxylic acid containing in the molecule mobile hydrogen atoms, to yield complex *compounds* of therapeutic value.
T. S. Wheeler.

Medicinal product. H. C. Fuller (U.S.P. 1,627,963, 10.5.27. Appl., 30.1.20).—Glycerol and derivatives, *e.g.*, glycerylchlorohydrin, can be employed in place of ethyl alcohol as solvents and preservatives in the preparation of medicinal mixtures. T. S. Wheeler.

5-Ethyl-5-*n*-hexylbarbituric acid. A. W. Dox, Assr. to Parke Davis & Co. (U.S.P. 1,624,546, 12.4.27. Appl., 15.12.24).—5-Ethyl-5-*n*-hexylbarbituric acid, m.p. 126° (cf. A., 1924, i, 1037), is claimed as a new substance of therapeutic value as a hypnotic. T. S. Wheeler.

Production of algin compounds. B. F. Erdahl (U.S.P. 1,625,301, 19.4.27. Appl., 20.7.26. Cf. Kunig; B., 1926, 784).—A colloidal solution of sodium alginate is added to dilute sulphuric acid, and after 24 hrs. the precipitate is washed, dried, and added to a solution obtained by the hydrolysis of beeswax with excess of ammonia solution. The product is a thick syrup, of value as a waterproofing agent for fibrous materials, *e.g.*, paper. The coated material is heated at 60°.
T. S. Wheeler.

Antidiabetic substance. F. S. Macy (U.S.P. 1,626,044, 26.4.27. Appl., 8.5.26).—Ground pancreas tissue is macerated with water, to which ammonium sulphate is then added to precipitate proteins. The liquid is filtered and treated with ammonia, the precipitate obtained being dried and mixed with fresh beef muscle juice. T. S. Wheeler.

Manufacture of the pure phosphorus-containing nuclear substance of milk casein. S. Posternak, Assr. to Soc. Chem. Ind. in Basle (U.S.P. 1,631,887, 7.6.27. Appl., 11.1.24. Conv., 25.1.23).—See E.P. 221,716; B., 1924, 966.

Preparation of products of organic origin. H. L. P. Tival (U.S.P. 1,630,985, 31.5.27. Appl., 24.4.22. Conv., 26.4.21).—See E.P. 179,164; B., 1923, 518 a.

Manufacture of physiologically-active substances from ovaries, corpus luteum, and placenta. M. Hartmann, Assr. to Soc. Chem. Ind. in Basle (U.S.P. 1,625,360, 19.4.27. Appl., 5.8.26. Conv., 8.5.23). —See E.P. 226,372; B., 1925, 149.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Colour photography and the "lignose screen." (Phot. Ind., 1927, 439—443).—The new lignose screen-unit process of colour photography on film is described in detail. The screen-film is prepared by the "dusting-on" process, using particles dyed in three colours having the following transmissions: red, λ5800 to λ7500 Å. with a maximum at about λ6400 Å.; green, λ5000 to λ6000 Å. with a maximum at about λ5500 Å.; blue, λ4000 to λ5200 Å., with a maximum at about λ4700 Å. The size of the screen elements ranges from about 0·005 mm. to 0·012 mm., the mean size being about 0·009 mm., *i.e.*, 10,000—12,000 elements per $mm.^2$ The proportion of coloured particles is 100 green, 70 to 80 red, and 80 to 90 blue. After the particles have been allowed to settle on the film coated with adhesive, the excess is removed and those adhering are rolled so as to spread them into contact at the edges. No filling agent is needed. The surface is finally varnished to protect the screen from the action of the developer and the reversing bath. The finished film transmits 16—17% of the incident light. The film is treated to prevent any possibility of separation of the emulsion from the screen, and the screen is not affected by dilute alcohol. A fine-grained, panchromatic, contrasty emulsion of speed 14 Scheiner is used, but in daylight the exposure of the screen film under a suitable filter is 40—75 times that required for a black-and-white reproduction on an emulsion of speed 14 Scheiner. Conditions for correct colour rendering, and the applications of the film are discussed. It can be used in all conditions in which ordinary screen-plates are employed, and in addition it possesses all the advantages of films, including their use in kinematography. W. Clark.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPTEMBER 30, 1927.

I.—GENERAL; PLANT; MACHINERY.

Heat transmission by radiation from non-luminous gases. H. C. Hottel (Ind. Eng. Chem., 1927, **19**, 888—894).—Coefficients of heat transmission based on the assumption of convection as the controlling mechanism cannot be satisfactory except for the apparatus in which they are determined, as radiation is neglected. When analysed the infra-red radiations from gases occur in bands, three each for carbon dioxide and water vapour. The calculation of heat quantities transmitted from gas mixtures containing these two gases is discussed and equations and curves are developed which enable them to be calculated with approximate accuracy from the gas and surface temperatures, gas partial pressures, shape factors, and the "black-body coefficient." The case of a flue etc. with change of temperature along the length is also considered.

C. Irwin.

Critical analysis of equations for the design of fractionating columns. L. H. Shirk and R. E. Montonna (Ind. Eng. Chem., 1927, **19**, 907—911).—The distillation of a mixture of ethyl alcohol and water in a column of nine plates of the bubbling hood type was studied. The relation of the actual number of plates used to the theoretical number as calculated by various methods to give similar results, termed the "column efficiency," was calculated. This gives a criterion by which the methods of calculation can be checked. The graphical stepwise method of McCabe and Thirle was found most satisfactory. Methods assuming continuous rather than stepwise change were markedly erroneous for such a column. The composition of the alcohol–water mixtures was determined by the immersion refractometer. Dilute mixtures (below 0·5% of alcohol) were determined by concentration by distillation and comparison with a prepared dilute mixture.

C. Irwin.

Suction of acid fumes from fume-cupboards. K. Veselý (Chem. Listy, 1927, **21**, 359—360).—Suction is produced by forcing air through an injector into a broader tube, thereby causing a partial vacuum which is in connexion with the fume-chamber. Corrosion of ventilating mechanism is thus avoided.

R. Truszkowski.

Industrial photo-colorimeter T.C.-B., independent of the eye. R. Toussaint (Bull. Soc. d'Encour., 1927, **126**, 421—429).—The colour to be measured is successively submitted to light passing through various coloured filters, and the intensity of the reflected light in each case is recorded by means of a photo-electric circuit. The curve obtained by plotting intensity against wave-length is characteristic of the colour. The relative brilliance of colours may also be assessed by measuring their true reflection of white light, allowance being made for difference in colour of the reflecting surface.

S. S. Woolf.

Rapid counter-current mixer. Kalinowsky-Stier (Chem.-Ztg., 1927, **51**, 630—631).—A description of the Eirich mixer, the cylindrical body of which rotates in the opposite direction to an eccentrically placed vertical shaft carrying inclined and staggered mixing arms.

S. I. Levy.

Sources of error in glass volumetric apparatus. H. V. E. M. Renn (J. Amer. Ceram. Soc., 1926, **9**, 850—859).

Patents.

Apparatus for the quantitative analysis of substances with the aid of Röntgen or cathode rays. F. Dessauer, Assee. of Metallbank & Metallurgische Ges. A.-G. (E.P. 252,207, 17.5.26. Conv., 15.5.25).—Apparatus for the quantitative analysis of substances by means of Röntgen or cathode rays dependent upon the observation of characteristic discontinuities in the ionisation–voltage curve in a chamber containing the substance, comprises an ionisation chamber in which the substance is arranged in the path of the rays, and a meter for registering the ionisation by reflecting a beam of light on to a drum coupled to a regulator for varying the voltage applied to the tube emitting the Röntgen or cathode rays.

J. S. G. Thomas.

Separation of solid materials of different specific gravities. G. Raw (E.P. 274,578, 30.4.26 and 26.2.27). —An even bed of the material, in such a state of subdivision that it offers resistance to air, is disposed on a pervious table with upstanding sides, and a static condition of air pressure, greater beneath the table than above it, is maintained so as to produce a mechanically fluid state and to separate the materials by density action. Stratification is increased by synchronising sharply defined pulsations of air and vibration of the table. The table may be inclined and scrapers may be provided to assist the discharge of material of lesser density.

W. G. Carey.

Roller mills for grinding and mixing. G. Dörstling (E.P. 274,784, 18.5.27).—Uniform pressure is applied to all the rollers of a roller mill by a single weighted lever system operated by a hand lever which acts directly on one of the rollers, the movement being transmitted to the other rollers by carriers. The rollers can be separated from one another to a variable extent

when mixing rather than grinding chocolate, pigments, etc., such separation being shown on an index.
W. G. CAREY.

Grinding bodies for ball, drum, and tubular mills. O. KORDT (E.P. 274,786, 26.5.27. Addn. to E.P. 261,264).—Grinding bodies for mills consist of globular steel bodies having not more than five flattened surfaces to intensify the beating, rocking, and rolling motions, and preferably any two of these surfaces are parallel to each other. W. G. CAREY.

Centrifugal apparatus for treating gases with liquids. L. and F. H. E. THEISEN (E. THEISEN) (E.P. 270,340, 30.4.27. Conv., 3.5.26. Addn. to E.P. 265,120).—The annular rotary plates are swaged or stamped to produce thereon annular series of protuberances each in the form of a truncated cone or provided with opposed walls which are segments of truncated cones.
H. HOLMES.

Drying agent for gases. I. G. FARBENIND. A.-G. (E.P. 272,173, 25.3.27. Conv., 7.6.26).—A solution of phosphorus pentoxide in 100% phosphoric acid is used for drying gases even when they are hot, and is employed, *e.g.*, for drying air for the combustion of phosphorus or gas mixtures containing phosphorus in the manufacture of phosphorus pentoxide. Phosphoric acid is obtained as a by-product, by using the drying mixture until all the phosphorus pentoxide is used up.
W. G. CAREY.

Heat-exchange apparatus. R. L. MUNDAY (U.S.P. 1,639,051, 16.8.27. Appl., 21.12.26. Conv., 5.12.25).—See E.P. 255,711; B., 1926, 729.

Separation of the elements of air or of other gaseous mixtures by liquefaction and rectification. J. LE ROUGE, Assr. to SOC. L'AIR LIQUIDE (SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE) (U.S.P. 1,638,005, 2.8.27. Appl., 21.7.22. Conv., 12.8.21).—See E.P. 184,454; B., 1922, 859 A.

Apparatus for heating, cooling, or drying materials. V. CANO (Re-issue 16,699, 9.8.27, of U.S.P. 1,592,078, 13.7.26).—See B., 1926, 808.

Furnaces (E.P. 257,261).—See X.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Siberian boghead coals. G. STADNIKOV (Brennstoff-Chem., 1927, **8**, 244—245).—Three samples of Siberian boghead coals contained roughly 1·3—4·0% of water, 5·9—14·8% of ash, and 70·4—81·0% of volatile matter (on the dry coal), 0·7—3·1% of sulphur and 0·6—1·1% of nitrogen (on the dry and ash-free coal). Their gross calorific value was 7584—8325 kg.-cal. On low-temperature carbonisation they yielded 39·1—48·8% of tar, with semi-coke containing 12·8—39·2% of ash and 11·9—14·9% of volatile matter. Analyses of the low-temperature tars are mainly remarkable for the low content of phenols (1·3—4·0%) and bases (0·2—0·9%) in the distillate up to 350°.
W. T. K. BRAUNHOLTZ.

So-called "algæ" of boghead coal. HELLMERS and POTONIÉ (Z. angew. Chem., 1927, **40**, 895—897).—The products obtained from boghead coal by maceration with Schulze's mixture, and named "algæ" by Bertrand and others, show no membranous structure. Aggregates of similar nature can be prepared by suitably introducing molten paraffin into aqueous gelatin solution, and are due to balling together of small drops. The phenomenon could be explained by a coagulation of bituminous constituents of the coal. A. COUSEN.

Oxidation of different coals at different temperatures. II. D. J. W. KREULEN (Brennstoff-Chem., 1927, **8**, 241—244).—When a flaming gas coal, a gas coal, a fat coal, or a lean coal is heated for several hours in air at 125°, 150°, 175°, or 225°, the resulting oxidation causes an increase in the fixed moisture content and a parallel increase in the humic acid content, the effect being greatest for the flaming gas coal and least for the lean coal, and being greater with increased temperature. The coking properties are reduced, 1 hr.'s oxidation at 175° being sufficient completely to destroy them. Even at 100—105° the coking properties are impaired, but heating at 175° in an atmosphere of carbon dioxide does not affect them. By prolonged heating of a flaming gas coal in air at 175°, and repeated extraction of the humic acid formed, 95% of the coal is converted into humic acid. W. T. K. BRAUNHOLTZ.

Effect of weathering on the softening and solidification points of coal. T. E. LAYNG and A. W. COFFMAN (Ind. Eng. Chem., 1927, **19**, 924—925).—Nitrogen was passed through a heated mass of 20—60-mesh coal and the back pressure due to softening measured. Tests with coal naturally weathered or oxidised by heating at 110° for various lengths of time showed that weathering raises the temperature of initial plasticity, decreases the maximum pressure developed, and decreases the temperature of coke formation. The curves obtained may therefore be used to indicate the extent of weathering. Coal cannot be preheated above 150° in air without injuring its coking properties, but in some cases higher temperatures in nitrogen were not harmful. C. IRWIN.

Influence of ash content on the calculated calorific value of ash-free coal. II. R. STUMPER (Brennstoff-Chem., 1927, **8**, 261—262; cf. B., 1927, 130).—The calorific value of the ash-free coal in mixtures of coal and calcium sulphate, calculated from the calorific value found by the bomb method and from the actual content of non-combustible matter, is constant until the gypsum content exceeds 20%, above which it rapidly falls. Calcium sulphate loses combined water and also suffers dissociation at 800°. The ash content experimentally determined is therefore less than the actual content, and the calorific value of the ash-free coal, calculated from the experimental ash content, steadily decreases with increasing content of gypsum.
W. T. K. BRAUNHOLTZ.

Utilisation of marine-animal and fish oils in motors. G. LUMET and H. MARCELET (Compt. rend., 1927, **185**, 418—420).—Marine-animal and fish oils have, in general, been found satisfactory as fuels in Diesel or semi-Diesel engines. The behaviour of the engine is more flexible than with gasoline, and the power developed is approximately the same. High viscosity at low temperatures presented the only difficulty.
C. W. GIBBY.

Determination of corrosive sulphur in motor benzol. R. Kattwinkel (Brennstoff-Chem., 1927, **8**, 259—260).—The corrosive ("active") sulphur is the difference between the total sulphur in the benzol before and after treatment with a metal such as copper or mercury. The total sulphur is determined by Schenk's method (cf. B., 1914, 130), a fresh sample of the benzol being then heated for 1 hr. under a reflux condenser with finely-divided copper bronze to remove the corrosive sulphur. An alternative method is to decompose the metallic sulphide with acid and determine the hydrogen sulphide liberated. W. T. K. Braunholtz.

Illuminating gas from lignite by processes employing internal heating by inert gases. F. Seidenschnur (Gas- u. Wasserfach, 1927, **70**, 728—736).—Low-temperature carbonising processes employing internal heating by gases have the advantages of more efficient and uniform heating and less decomposition of tar and semi-coke. The Limberg and Lurgi processes are not suitable for supplying illuminating gas from lignite, since the gas produced is used up for heating purposes in the processes themselves. A more promising method is carefully to dry screened or briquetted lignite by heating it in air of gradually diminishing humidity, to distil the dried product by internal gas heating at 420—440°, and finally to degasify the semi-coke at red heat. The gas produced at 440° (83 litres/kg. of dry lignite) has, after removal of benzine, a gross calorific value of 1660 kg.-cal./m.^{3}, contains 73·5% CO_2, and may be utilised for drying the raw lignite. The corresponding semi-coke, containing 16·1% of ash and 23·4% of volatile matter, yields, when heated to redness, a gas (266 litres/kg.) containing 12·8% CO_2, and a porous and easily combustible coke with 4% of volatile matter and a nett calorific value of 6340 kg.-cal. The carbon dioxide-free gas contains 26·0% CO, 51·5% H_2, 20·8% CH_4, 0·8% N_2, and has d 0·418 and gross calorific value 4480 kg.-cal., thus resembling ordinary coal gas. The yield of low-temperature tar in the above process is 100—110% of that found by the Fischer assay, the loss due to decomposition being only 1·5%. By cracking, this tar could be converted into benzine and a gas of high calorific value. Large-scale tests of the above process have not yet given results equal to those above quoted, obtained in laboratory experiments.

W. T. K. Braunholtz.

Low-temperature distillation of long-flame coals. A. Léauté (Compt. rend., 1927, **185**, 465—467).—The influence of the binding material upon the distillation of long-flame coal has been studied. Porosity of the residue can be avoided by using as a binding material anthracene oil, which has a minimum content of tar. The residue is then hard and compact, similar to anthracite, and can be used as such. A quantity of oil is obtained about equal to that of the binding material used, and 2—15% of gas. C. W. Gibby.

Evaluation of water-gas oil. T. Mitsuhashi (J. Fuel Soc. Japan, 1927, **6**, 2—3).—The oil is fed into the top of a vertical porcelain tube, 3 cm. in diam. and 60 cm. long, filled with broken firebrick and heated in an electric furnace. The apparatus is first filled with carbon dioxide, and again swept out with carbon dioxide at the end of the experiment, the gases being passed through potassium hydroxide on their way to a gasometer. The yield and quality of the gas produced at any given temperature are determined.

W. T. K. Braunholtz.

Eislingen shale oil. K. Neubronner (Petroleum, 1927, **23**, 789—796).—The shale was carbonised in a rotary cylindrical retort, yielding 6—6·4% of crude oil and 2·9%, or 35 litres, of gas per kg. The net calorific value of the shale was 1380. The crude oil had d 0·900—0·920, open flash pt. 20°, wax content 0·45—0·50%, sulphur content 2·3—3·5%, and contained about 0·75% of aromatic hydrocarbons. The distillation test gave 30—34% at 200° and 63—71% at 300°. When the crude oil was distilled in an atmosphere of nitrogen, and the fraction up to 150° repeatedly redistilled, 10·6% of the crude oil distilled over up to 125°, and 6·4% from 125° to 150°. The fractions up to 74° remained colourless, whilst those up to 125°, though colourless at first, became dark and gave a reddish-violet deposit on keeping. The benzine fraction 60—180° had d 0·790—0·795, sulphur content 3·5—4·0%, and polymerised readily with deposition of asphaltic substances. In refining the 150° fraction the diolefines were removed by acid and soda treatment. Refining with about 5% of sulphuric acid and 1—1½% of 15% soda solution gave a spirit with passable smell. Better results were not obtained with sodium plumbite solution. The refined benzine had d 0·780—0·790, n_D^{20} 1·4500—1·4540, mercury acetate value 300—340, sulphur content 2·55—2·65%. A commercially feasible method of desulphurising the benzine was not found. H. Moore.

"Pinking" in internal-combustion engines. G. B. Maxwell (J. Inst. Petrol. Tech., 1927, **13**, 224—243).—The researches and opinions of other workers on pinking, detonation, and flame phenomena are reviewed. The distinction between pre-ignition and pinking and the effect of various anti-knock and knock-inducing compounds are discussed, as also the effect of carbon disulphide on the lag period, and of the shape of the combustion chamber and engine details on pinking.

H. Moore.

Effect of metallic vapours on the ignition of substances. A. Egerton and S. F. Gates (J. Inst. Petrol. Tech., 1927, **13**, 244—255).—The temperature of ignition of petrol in a Moore apparatus was raised by 160° by the addition of 0·25% of lead tetraethyl, and by 145° when the vapour of that compound was admitted to the crucible. Lead oxide, produced as a fine dust by sparking between lead electrodes, only caused a slight rise in ignition temperature, but argon passed over a low-tension arc between lead terminals and directly introduced into the crucible caused a rise of 60°, or 70° with a similar arrangement using nitrogen. The rise for a given quantity of lead was of the same order as when lead tetraethyl was added to the petrol. A table is given of elements effective or ineffective when tested by this method. The effect of the metal was found to be similar to that of its organo-derivative. The metal itself in a state of incipient oxidation is the effective agent. Among the rises of temperature noted with a concentration of 0·1% of the substance tested are selenium

diethyl 140°, iron carbonyl 130°, bismuth triethyl 120°, lead ethyl 90°, nickel carbonyl 40°, tellurium diethyl 55°, and bismuth triphenyl 42°. The effects of lead tetraethyl on the ignition temperatures of different combustibles are as follows. Normal alcohols are little affected; aldehydes, very greatly raised; methyl*cyclo*hexane, more affected than *cyclo*hexane; benzene, effect only slight; and *iso*hexane, less affected than *n*-hexane. Anti-knock compounds diminish the rise in temperature by hindering oxidation of the fuel. H. Moore.

Significance of igniting temperatures. A. Egerton and S. F. Gates (J. Inst. Petrol. Tech., 1927, **13**, 256—272).—The ignition point is defined as that at which the rate of gain of heat by a volume element of vapour balances the rate of loss of heat, and the temperatures given in ignition testers similar to that of Moore are termed "igniting points." The results of different investigators on igniting points are reviewed and shown to be tolerably concordant. The authors' experiments were conducted with an iron pot 1½ in. diam. and 2¼ in. high, heated air being introduced at the rate of 330 c.c. per min. and temperatures measured by a thermo-couple near the bottom of the pot, the surface of which was polished. The size of the drop did not greatly affect the igniting temperature, nor did the rate of air supply, so long as this was adequate. Oxidation of the surface of the pot caused a rise in igniting temperatures. Differences of material of the surface had considerable influence on the temperature, especially in the case of acetone. An increase of surface by the introduction of iron plates or glass wool into the pot gave higher igniting temperatures, flameless combustion occurring before ignition. The introduction of a halide lowered the temperature. The introduction of ions from radium bromide did not influence the temperature. Aldehydes ignite at lower temperatures than the corresponding alcohols and hydrocarbons. The igniting temperatures are related to the knocking tendencies of fuels in engines. H. Moore.

Effect of certain organic compounds on the igniting and "knocking" characteristics of petrol. A. Egerton and S. F. Gates (J. Inst. Petrol. Tech., 1927, **13**, 273—280).—The effects of various purely organic compounds on the knocking tendencies of petrol were studied in connexion with their influence on the igniting temperature. *m*-Xylidine is the most effective anti-knock of the arylamines, but is 30 times less effective than lead tetraethyl. Aldehydes, acids, nitro-compounds, nitrates, etc. promote knocking; *m*-compounds are rather more effective than *o*- and *p*-compounds, and diethylamine is less effective than diphenylamine. Quinoline, pyridine, and carbylamine are ineffective in comparison with aniline. Ethyl iodide, benzoquinone, cresol, phenol, and diphenyl ether are effective, but less so than aniline. The comparative effects of iron carbonyl, lead ethyl, nickel carbonyl, aniline, *m*-xylidine, and toluene are respectively 600, 400, 160, 11, 12, and 1. Alcohol and benzene act as diluents, raising the H.U.C.R. according to the quantity added; 50% of carbon disulphide raises the H.U.C.R. 6%, in agreement with its influence on igniting temperatures. Ether destroys the influence of lead tetraethyl, in accordance with its influence on igniting temperature. Aniline raises the igniting temperature of acetaldehyde by 110°. The addition of hydrocarbons of high mol. wt. depresses the H.U.C.R. disproportionately. No substance effective on the engine did not affect the igniting point, but the reverse does not always hold. The molecules of lead tetraethyl and aniline are effective on the igniting point in the proportion of 70 : 1. A table showing the effect on igniting point of various organic compounds is given. H. Moore.

Theories of anti-knock action. A. Egerton and S. F. Gates (J. Inst. Petrol. Tech., 1927, **13**, 281—299).—Anti-knock compounds are negative catalysts which remove the substances which autocatalyse combustion, generally peroxides. Such metallic compounds form comparatively stable peroxides which destroy fuel peroxides and are then regenerated. The organic anti-knock compounds behave in a similar manner, but themselves undergo combustion. The anti-knock effect implies the slowing down of the initial stage of combustion, is chemically the same as the effect on igniting temperatures, and suppresses detonation, even when the fuel and the anti-knock compound are both in the gaseous state. The active constituent of an organo-metallic anti-knock compound is the metal atom, and this must be oxidisable. Rise of igniting temperature and anti-knock effect are attended by diminution of the rate of oxidation of the fuel. The effective organic compounds are amines, quinones, phenols, etc. with high b.p. and igniting temperatures. Anti-knock compounds affect the combustion of aldehydes, but hardly that of alcohols. The chemistry and physics of the action of the compounds are discussed. H. Moore.

Anti-knock dopes. R. A. Weerman (J. Inst. Petrol. Tech., 1927, **13**, 300—307).—The spontaneous ignition temperatures of a number of organic and organo-metallic compounds were tested in an apparatus consisting of an iron pot roughly polished inside, at the bottom of which air is passed at the rate of 330 c.c. per min., a drop (0·013 c.c.) of the liquid to be tested being allowed to fall through a hole in the cover of the pot while the temperature is rising at intervals of 10° and 5°. The effect of the addition of various small percentages of different compounds to a fuel was tested, and it was found that the more notable anti-knock compounds raised the spontaneous ignition temperature by an amount of the order of 105° to 170°. Compounds which raised this temperature had an anti-knock effect in the engine and *vice versâ*, though not in exact proportion. An anti-knock compound must be volatile and capable of reduction during the compression stroke in the engine. Silicon tetraethyl lowers the spontaneous ignition temperature and increases knocking. On condensing the products of combustion from the apparatus it was found that the addition of 2 g. per litre of lead tetraethyl to petrol caused less water to be formed and a reduction of the acid value of the oil layer, the consumption of oxygen being also reduced. Importance is to be attached to the reactions which occur during the compression stroke in an engine. H. Moore.

Fuller's earth and its uses in the petroleum industry. A. Rauch (J. Inst. Petrol. Tech., 1927, **13**, 325—330).—Fuller's earth has an important action in neutralising acid oils, thus saving the expenses of soda

washes and the risk of emulsion formation. The earth itself has generally an acid reaction, and acid earths have the better bleaching properties, though not in proportion to their activity. The deodorising action of fuller's earth in vapour-stage refining is attributed to the polymerisation and adsorption of unsaturated and sulphur compounds. On opening the filter presses in which fuller's earth has been separated from edible oils, fires sometimes occur owing to rapid oxidation in contact with air.

H. Moore.

Action of sulphuric acid on unsaturated and aromatic hydrocarbons. W. R. Ormandy and E. C. Craven (J. Inst. Petrol. Tech., 1927, **13**, 311—320).—The assumption that shaking with 98% sulphuric acid completely removes aromatic and unsaturated hydrocarbons is erroneous, a large proportion of the olefines being separated as saturated hydrocarbons, probably open-chain paraffins. Alkyl hydrogen sulphates only are formed in small quantities from some unsaturated compounds, and a large proportion of aromatic sulphonic acids disappears when olefines are present. The barium carbonate method carried out in the cold gave very accurate results in determining aromatic compounds. In mixtures of a turpentine fraction and aromatic-free petrol and the same petrol with added aromatic compounds the loss to sulphuric acid was about equal to the turpentine plus aromatics, but when amylene was substituted for turpentine the results only corresponded to about one half of the unsaturated compounds. The conductivity of the final liquor was adopted as criterion of the sulphonic acids present, and gave very accurate figures for the aromatic compounds present in a mixture. In the absence of olefines aromatic compounds may be determined by the amount of sulphonic acids formed on shaking with 98% sulphuric acid, but in the presence of olefines aromatic compounds are not entirely removed, partly condensing with the hydrocarbons formed from the olefines. The sulphuric acid wash from amylene contains very little amyl hydrogen sulphate.

H. Moore.

Carbon ratio. M. Stuart (J. Inst. Petrol. Tech., 1927, **13**, 308—310).—It is pointed out that the carbon ratio, often used as a guide to the character of oils found in the vicinity of coal, or as an indication of the degree of probability of finding petroleum oil in a given district, is often erroneously described as the carbon/hydrogen ratio, as if it were the ratio of these elements found by ultimate analysis. It is actually the percentage ratio of fixed carbon to the total of fixed carbon and volatile matter found by proximate analysis in a coal. This ratio in certain coals may be 50, whilst the same coals contain more than 90% of carbon. H. Moore.

Economy of low-boiling distillate of fuel gasoline. Y. Tanaka (J. Fuel Soc. Japan, 1927, **6**, 39—40).—The volatility of a gasoline poor in the lower-boiling fractions, *e.g.*, one from which a low-boiling fraction has been removed for solvent or other uses, can be increased by the addition of alcohol, which forms minimum-boiling azeotropic mixtures with the gasoline hydrocarbons. From a study of such mixtures the following formulæ have been established: $W_H = (160 - T_H)/1{\cdot}15$ for paraffins and naphthenes, $W_H = (140 - T_H)/0{\cdot}89$ for aromatic hydrocarbons, and $(T_H - T_{AZ})\ (81 - T_{AZ}) = K$, where T_H is the b.p. of the hydrocarbon and W_H its percentage by weight in the azeotropic mixture, of which T_{AZ} is the b.p. K is about 236 for paraffins and naphthenes, and about 150 for aromatic hydrocarbons. A. B. Manning.

Preparation of benzene by polymerisation of acetylene. S. Iki and M. Ogura (J. Soc. Chem. Ind. Japan, 1927, **30**, 461—469).—Acetylene was passed through a hard glass U-tube containing activated carbon of many kinds. There is little difference in catalytic activity between activated charcoal, wood charcoal, coke, and coalite. A special relationship is not found between the decolorising action and the catalytic action of the carbons. The coarse granular catalysts are suitable for the purpose; those in a fine state are apt to cause local heating in the reaction tube. The optimum temperature is 650—660°, and gas velocity 10 litres per hr.; the yield of the oil is then 60—70% of the acetylene used, and it contains 50—60% of light oil distilling below 150°, composed mainly of benzene. Under constant conditions the results of the experiments are almost similar for all catalysts. K. Kashima.

Crystallisation of paraffin wax. F. H. Rhodes, C. W. Mason, and W. R. Sutton (Ind. Eng. Chem., 1927, **19**, 935—938).—Paraffin wax was fractionally melted in a thermostat, the mol. wt. of each fraction determined, and its behaviour on solidification studied microscopically. All the fractions behaved in a similar way. Small plates were first formed, but with fairly rapid cooling needles then developed. With very slow cooling plates only were formed. The two forms are therefore of identical composition, and are not even allotropic modifications, as they may co-exist at constant temperature. The needles, as is shown by examination under crossed Nicols, are really plates tightly rolled up. The plates may be hemimorphic, and the rolling caused by differences in surface tension. C. Irwin.

Structure of the asphalt micelle. F. J. Nellensteyn (Chem. Weekblad, 1927, **24**, 414—421).—The disperse phase consists of carbon particles which adsorb and are surrounded by heavy protective hydrocarbons and their dissociation products, forming micelles in the medium. Asphalt shows analogies with such highly-protected organosols as sodium chloride in benzene.

S. I. Levy.

Production of oil from bituminous coal. Work of the Fuel Research Station. C. H. Lander (J. Inst. Petrol. Tech., 1927, **13**, 151—168).

Refining dry-cleaners' solvent. Flowers and others.—See V.

Ammonium sulphate. Terres and Schmidt.—See VII.

Determination of carbon monoxide. Kast and Schmidt.—See VII.

Refractories for oil-gas manufacture. Knollman.—See VIII.

Patents.

Gas-producer and combined furnace. T. R. Wollaston (E.P. 274,286, 3.8.26).—The furnace described in E.P. 243,092 is modified to provide ready

access to the ports and burners supplied with secondary air, means for closing these ports and burners independently, as by detachable firebrick plugs, and means for controlling the actual and relative amounts of primary and secondary air supplied. The dampers or valves controlling the primary and secondary air supplies are positioned from a common lever with provision for independent adjustment of the effective leverages. When applied to a boiler furnace, water may be circulated between the producer jacket and the boiler, or water preheated in the jacket may be pumped into the boiler. Hot water from the jacket may be atomised at the primary air inlet. H. Holmes.

Coking retorts. Collin & Co., and J. Schaefer (E.P. 266,286, 23.9.26. Conv., 20.2.26).—The coke discharged from any one of a row of retorts is conveyed selectively to any one of several cooling chambers in front of the retorts across a platform movable transversely to the retorts and arranged on a level with the discharge openings of the retorts and the charging openings of the chambers. The platform may be a rotary disc provided with a stripper, adjustable rotarily for directing the coke into the selected chamber, or it may be provided with a stripper or a base, such as an endless conveyor band, movable parallel to the axes of the retorts. A container wall arranged around the platform may be provided with closable openings opposite the retorts and the chambers and, if desired, with quenching sprayers. H. Holmes.

Coke ovens. N. V. Silica en Ovenbouw Mij., Assees. of C. Otto & Co. G.m.b.H. (E.P. 268,832, 4.4.27. Conv., 3.4.26).—Each inspection opening in the top of the oven over a vertical heating flue is provided with a heat-insulating block and a plugged covering block, both removable. The heat-insulating block is seated on an annular shoulder, and is apertured in line with the plugged aperture in the cover block. A distance tube, *e.g.*, of brick or iron covered with asbestos, may be provided between the blocks. H. Holmes.

Regenerative coke ovens. Simon-Carves, Ltd., and J. H. Brown (E.P. 274,653, 6.8.26).—The vertical heating flues of a regenerative coke oven are divided into four sections, two up- and two down-flow flues, each pair of down-flow flues being connected to a regenerator placed between adjacent oven walls. The horizontal flue connecting the vertical flues is divided into two halves, which are each tapered from the centre to each end, and thus allow the vertical flues to extend to a higher level. H. D. Greenwood.

Production of active carbon. K. S. C. Bone, and Wilson Brothers Bobbin Co., Ltd. (E.P. 274,538, 19.3.26).—Finely divided wood, impregnated with calcium acetate, is fed by means of helically-grooved internal conveyors through one or two retorts, in which it is gradually heated to 1000°. As a result of the progressive heating creosote oil is produced instead of the usual wood tar distillates, and this together with the decomposition products of calcium acetate are withdrawn below 450°. H. D. Greenwood.

Dissociation of carbonaceous gases or vapours. H. E. Potts. From Canada Carbide Co., Ltd. (E.P. 274,573, 24.4.26).—In a continuous process for the manufacture of carbon black by the dissociation of hydrocarbon gases in a special vertical retort, the composition of the gas is such that the heat of reaction is sufficient to maintain the required dissociation temperature. The gases are introduced into the retort without preheating in order to prevent polymerisation, and suddenly heated to dissociation temperature. The liberated hydrogen is burnt from the base of the retort, the carbon being collected in flues with baffles and continuously removed by a screw conveyor. H. D. Greenwood.

Motor fuel. C. E. H. North and A. H. Hudson (U.S.P. 1,637,007, 26.7.27. Appl., 5.8.25).—Detonation is suppressed by adding 0·05—1·5% by vol. of carbon tetrachloride to motor fuel. C. O. Harvey.

Gaseous fuel [for welding etc.]. J. Harris, Assr. to J. R. Rose (U.S.P. 1,637,187, 26.7.27. Appl., 28.5.23).—A mixture of acetylene with 5—20% by vol. of benzine vapour is used. C. O. Harvey.

Recovery of benzene from coal gas. N. V. Silica en Ovenbouw Mij., Assees. of C. Otto & Co. G.m.b.H. (E.P. 265,984, 11.2.27, Conv., 12.2.26).—The loss of benzene in the exhaust gas of the vacuum pump during the distillation of wash oil is reduced by (1) adding the exhaust gas to the coal gas before washing, (2) washing the exhaust gas in a separate washer, (3) extracting the benzene from the gas by an absorbing agent such as activated carbon or silica gel. H. D. Greenwood.

Low-temperature distillation or coking of fuels. J. Plassmann (E.P. 258,261, 7.9.26. Conv., 10.9.25).—The close packing of fuel, prior to distillation, is effected in a vertical retort by the jigging movements (without rotation) of a tapered plate suspended within the retort or by the horizontal movement of a vertical tapered plate arranged at one side of the retort. The shape of the shaking device may be such that it exerts a pressing action. H. D. Greenwood.

Treatment for transport and/or utilisation of bituminous and like materials. N. Testrup, T. Boberg, and Techno-Chemical Laboratories, Ltd. (E.P. 274,540, 20.3.26).—Insoluble matter is removed from Trinidad asphalt by treating the molten water-free asphalt with about an equal weight of a heated volatile solvent in a vertical tubular vessel. The solution overflows into a cylindrical settling tank with conical bottom from which the settled impurities are withdrawn, whilst the solvent is recovered by heating the mixture in a closed steam coil evaporator and finally with superheated steam. The hot purified asphalt can be transported in heated tank steamers or sufficient solvent can be left in the residue to render it fluid at ordinary temperatures. H. D. Greenwood.

Production of hydrocarbons and derivatives thereof from mineral oil or other bitumens and products obtained therefrom. I. G. Farbenind. A.-G. (E.P. 249,501 and 274,401, 8.3.26. Conv., [A] 19.3.25. Addns. to E.P. 247,583; B., 1927, 595).—The process described in the main patent can be applied to (A) crude mineral oils, shale oils, fractions or residues of such materials, ozokerite, mineral pitch, etc., (B) conversion products of mineral oils and bitumens, *e.g.*, cracked products, acid sludge, etc., in order to convert them

into valuable oils consisting principally of colourless, saturated benzines. A. B. Manning.

Production of hydrocarbons [from coal etc.]. I.G. Farbenind. A.-G. (E.P. 249,156 and 274,404, 13.3.26. Conv., 14.3.25; cf. E.P. 247,582—4; B., 1927, 595).—(a) Coal or similar carbonaceous material is treated with hydrogen under pressures of at least 50 atm. and at high temperatures (*e.g.*, under 200 atm. at 450°) with or without the aid of contact masses, and the tar-like products so obtained are further treated with hydrogen in the presence of catalysts under similar conditions of temperature and pressure. 70% or more of the carbon contained in the solid fuels is thereby converted into valuable hydrocarbons. (b) The tar vapours are subjected to the second stage of the treatment without condensing them intermediately. A. B. Manning.

Preparation of mixtures of carbon monoxide and hydrogen from hydrocarbons. L. Casale (E.P. 274,610, 2.6.26).—Gaseous hydrocarbons mixed with excess of steam are completely decomposed to hydrogen and carbon monoxide when heated above 1000° at reduced pressure. The heat absorbed in the reaction is supplied by admitting the required proportion of oxygen or air into the mixture. If air or air enriched with oxygen is used, the mixture of hydrogen and nitrogen required in the synthesis of ammonia is obtained. H. D. Greenwood.

[Cracking] treatment of petroleum. J. W. Lewis, jun., Assr. to Atlantic Refining Co. (U.S.P. 1,636,520, 19.7.27. Appl., 4.4.25).—The oil (in liquid phase) is heated and cracked while passing through a tube containing a helical spiral, the cross-section of the spiral stream of oil being but a small fraction of that of the tube. C. O. Harvey.

Process and apparatus for cracking hydrocarbons. C. Arnold. From Standard Development Co. (E.P. 274,763, 14.3.27).—Reduced crude oils are cracked so as to produce not more than 0·25% of sediment in the tar by heating the oil in a coil, and then passing it to an enlarged insulated vessel, where cracking is completed under pressure. The cracked product is run at reduced pressure to a tubular still, fitted with a fractionating column, where it is further heated to vaporise at least 30% of the contained gas oil. The tars obtained from the cracking of the gas oil and from the reduced crude oil are mixed for fuel oil. H. D. Greenwood.

Process and apparatus for the conversion of heavy hydrocarbon oils into lighter oils. V. W. Northrup (E.P. 275,120, 21.3.27).—Increased yields of gasoline are obtained by mixing the vapours from heavy hydrocarbon oils with hydrogen or a gas containing hydrogen (natural gas) and subjecting the mixture to an alternating current field of 2000—8000 volts to effect ionisation. Thence the vapours pass through a catalysing tube at temperatures of about 385—538°, a suitable catalyst being nickel containing 5% Fe and 5% Al. After passage through a dephlegmating column the vapours are partly condensed in a tubular cooling chamber maintained at 93—204°, the condensate being returned for retreatment and the uncondensed vapours passing to the condenser. In order to obtain gasoline of satisfactory boiling range etc., a battery of catalysing tubes maintained at different temperatures is used in conjunction with each still. C. O. Harvey.

Apparatus for the solidification of liquid hydrocarbons. H. Neveu, Assr. to Y. de Paniagua (U.S.P. 1,636,644, 19.7.27. Appl., 27.9.24. Cf. U.S.P. 1,525,409; B., 1925, 276).—The apparatus comprises a vessel fitted with mixing arms provided with conduits and attached to two vertical shafts, the one shaft serving for the admission of the liquid hydrocarbon and an emulsifier and the other shaft for the admission of the hardening agent. C. O. Harvey.

Distilling [oils] with aluminium chloride. A. McD. McAfee, Assr. to Gulf Refining Co. (U.S.P. 1,636,144, 19.7.27. Appl., 20.7.25).—In a cracking distillation process employing aluminium chloride the oil is subjected to a preliminary purifying distillation with exhausted aluminium chloride sludge. C. O. Harvey.

Distillation of crude oils. F. Tinker (E.P. 274,959, 28.4.26).—A heat-interchange arrangement wherein the recovery of outgoing heat from various parts of the system is facilitated by delivering hot liquids or vapours or liquid–vapour mixtures into a mixing chamber, where they attain a common temperature prior to their entering a chamber for separating liquid and vapour. C. O. Harvey.

Decolorising, clarifying, and purifying petroleum oils. H. L. Kauffman and I. A. Clark, Assrs. to H. L. Kauffman (U.S.P. 1,636,938, 26.7.27. Appl., 11.11.24. Renewed 25.2.27).—Untreated, finely-divided clays, containing enough water to render them sufficiently fluid for pumping through pipes, are mixed with the oil, the mixture being heated at a temperature not above the b.p. of the oil, and then steamed down to a temperature over 100°. The vapours removed with the spent steam are condensed. After further cooling, the oil is separated from the filtering medium. C. O. Harvey.

Purification of oils. F. W. Weber (U.S.P. 1,636,946, 26.7.27. Appl., 18.3.26).—After refining with sulphuric acid and separation of the acid sludge, the acid–oil mixture is treated with a colloidal solution containing basic chlorides of iron and rare-earth metals, whereby ferric hydroxide is precipitated. The further addition of a practically saturated solution of sodium silicate produces a voluminous heavy precipitate of silicon and rare-earth metal hydroxides, and, after settling and separating operations are completed, the oil is distilled. C. O. Harvey.

Retort oven for low-temperature carbonisation. P. C. Zuyderhoudt (U.S.P. 1,636,975, 26.7.27. Appl., 27.9.24. Conv., 22.10.23).—See E.P. 229,880; B., 1925, 345.

III.—TAR AND TAR PRODUCTS.

Naphthalene formation in coal tar. Y. Kosaka and Y. Oshima (J. Fuel Soc. Japan, 1927, **6**, 4—8).—Phenol and its homologues, when passed over coke at 850°, are decomposed to varying extents with formation of benzene, naphthalene, anthracene, and a gas comprising mainly hydrogen, carbon monoxide, and methane. Under the same conditions benzene and toluene yield

naphthalene, anthracene, and a gas comprising mainly hydrogen and methane. Methane is formed by demethylation of homologues of benzene and phenol, whilst carbon monoxide results from the decomposition of or action of carbon on phenols. It is suggested that naphthalene in coal tar is derived partly from the decomposition of alkylnaphthalenes and of naphthols and their derivatives, but mainly from the decomposition of phenols formed during the early stages of coal carbonisation. W. T. K. Braunholtz.

Determination of the softening point of pitch and asphalt by Kraemer and Sarnow's method. H. Mallison (Z. angew. Chem., 1927, **40**, 927—928).—The softening point of pitch or asphalt as determined by Kraemer and Sarnow's method depends on the rate of heating. Values 2—3° lower are obtained for the softening point when the increment of temperature is 1° per min. than when it is 2° per min. Klinger's modification of this method (B., 1914, 130) is recommended, the initial temperature of the water-bath being 20—25° below the expected softening point of the pitch.
R. Brightman.

Sulphur in motor benzol. Kattwinkel.—See II.

Action of sulphuric acid on hydrocarbons. Ormandy and Craven.—See II.

Patent.

Benzene from gas (E.P. 265,984).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Hydro-ψ-thiocyanic acid as a dye for cotton. B. Melis (Annali Chim. Appl., 1927, **17**, 346—351).—This acid (cf. A., 1927, 346) when dissolved in sodium hydroxide or sulphide dyes cotton a lemon-yellow colour. The dye is fast to light, washing, boiling, rubbing, hot ironing, the action of sulphur dioxide, ammonium acetate, 10% sulphuric acid, or 30% acetic acid. Alkali causes a slight deepening of colour, boiling sodium hydrogen sulphate solution a slight heightening; the dye is not, however, transferred to admixed undyed cotton. Washing with soap and sodium carbonate causes a slight modification, chlorine a slight bleaching, and mercerisation a modification of colour with transference to undyed cotton. The mechanism of the dyeing is discussed; it is attributed to the presence of SH- and S·S-groupings.
E. W. Wignall.

Patents.

Manufacture of compounds of aromatic *p*-diamines with sulphur dioxide. I. G. Farbenind. A.-G. (E.P. 269,583, 14.4.27. Conv., 17.4.26).—Aromatic *p*-diamines on treatment with sulphur dioxide in the absence of water (*e.g.*, by saturating their benzene solution with the gas) are converted into water-soluble compounds remaining unchanged in air, which are used as hair dyes, photographic developers, etc.
B. Fullman.

Reduction of aromatic nitro-compounds. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 274,562, 21.4.26).—In the reduction of aromatic nitro-compounds, finely-divided iron oxide of pigmentary value may be obtained by carrying out the reduction with iron and an acid-reacting concentrated solution of an easily soluble salt of a metal more electro-positive than iron, the acid being either an added non-reducing mineral acid or derived from the hydrolysis of the salt (of a non-reducing mineral acid). Easily soluble chlorides (*e.g.*, those of the alkali or alkaline-earth metals, magnesium, and zinc) are especially suitable, as well as ferrous chloride. A sludge of black ferrosoferric oxide or yellow to brown hydrated ferric oxide remains, which on sedimentation etc. may either be used as a pigment immediately or converted into a red shade by calcination. B. Fullman.

Manufacture of cyclic hydrocarbons and derivatives thereof. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 251,270, 19.4.26. Conv., 24.4.25).—Aromatic ketones having both a methyl or methylene group and a free hydrogen atom *ortho* to the carbonyl group condense internally to form anthracene derivatives when their vapours are passed over heated porous contact substances (silica gel, alumina gel, active charcoal, etc.); *e.g.*, phenyl *m*-xylyl ketone (active carbon at 380—400°) gives β-methylanthracene, whilst *o*-tolyl α-naphthyl ketone (aluminium oxide at 420°) gives naphthanthracene, m.p. 167° [Elbs (Ber., 1919, **52**, 2211) gives 141°]. Similarly, mixed aliphatic aromatic ketones yield derivatives of 5-membered rings; *e.g.*, *m*-xylyl methyl ketone (active charcoal) gives methylindene. B. Fullman.

Manufacure of colour lakes. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 274,627, 28.6.26).—Diphenylamine derivatives, made by condensing a halogenated aromatic nitro-compound with a primary or secondary arylamine which may contain carboxylic or sulphonic groups, are mixed with the usual lake components or, if acidic, are converted into insoluble barium or calcium salts. Thus sodium 2 : 4-dinitrodiphenylamine-2′-carboxylate and sodium carbonate are dissolved in water, barium sulphate and a solution of aluminium sulphate are added, and an orange lake is precipitated by addition of barium chloride solution. 2 : 4-Dinitrodiphenylamine-4′-sulphonic acid gives a greenish-yellow lake. C. Hollins.

Manufacture of [chrome] dyes of the triarylmethane series. I. G. Farbenind. A.-G. (E.P. 263,879, 3.1.26. Conv., 2.1.26).—The carbinols obtained by condensing substituted benzaldehydes with 2 mols. of *o*-cresotic acid are converted into soluble compounds by heating with sodium sulphite solution. The filtered solutions on evaporation give colourless products, which regenerate the carbinols on treatment with acid, and dye wool blue to violet and brown shades by afterchroming. Products derived from 4-*p*-tolylaminobenzaldehyde (violet), 4-(6′-chloro-*o*-tolylamino)benzaldehyde (violet), *p*-chlorobenzaldehyde (greenish-blue), 2 : 6-dichlorobenzaldehyde (reddish-brown), 2 : 6-dichloro-3-hydroxybenzaldehyde (brown), 2 : 4 : 6-tribromo-3-hydroxybenzaldehyde (brown), and hydroxynaphthaldehydedisulphonic acid (E.P. 19,498 of 1897; brownish-violet) are described. C. Hollins.

Manufacture of chloranil and bromanil. L. B. Holliday & Co., Ltd., and C. Shaw (E.P. 274,700, 26.11.26).—Chloranil results when *p*-nitrosophenol is chlorinated in 30% hydrochloric acid, the temperature

being raised slowly to 90°. Bromination of *p*-nitrosophenol in glacial acetic acid gives bromanil.
C. Hollins.

Manufacture of derivatives of the anthraquinone series. I. G. Farbenind. A.-G., Assees. of Farbenfabr. vorm. F. Bayer & Co. (E.P. 250,968, 17.4.26. Conv., 17.4.25).—4 : 8-Diaminoanthrarufin-2 : 6-disulphonic acid, when heated with rather more than 2 mols. of 40% aqueous formaldehyde at 95° for $1\frac{1}{2}$ hrs., gives the *NN'-dimethyl* derivative, precipitated by addition of concentrated hydrochloric acid. It dyes wool blue from an acid bath. One or both of the sulphonic groups may be removed, *e.g.*, by reduction with sodium hyposulphite, or by hydrolysis with 90% sulphuric acid and boric acid, especially in presence of a little stannous chloride. The *monosulphonic acid* (*aniline* salt prepared) gives a light greenish-blue on wool. Similar series of compounds are obtained from the 3 : 7-disulphonic acid and from 4 : 5-diaminochrysazin-2 : 7-disulphonic acid.
C. Hollins.

Manufacture of anthraquinone derivatives. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 274,211, 19.4.26).—The boric esters of 4 : 8-diaminoanthrarufin-2 : 6-disulphonic acid and its *N*-methyl derivatives condense in concentrated sulphuric acid below 20° with phenols or their ethers to give compounds, which when heated in dilute mineral acid or alkali (or in the cold with caustic alkali) are converted into 2-*p*-hydroxyaryl derivatives, with loss of the 2-sulphonic group. The remaining sulphonic group may be removed by known methods, *e.g.*, by reduction with sodium hyposulphite. 4 : 8-Diaminoanthrarufin-2 : 6-disulphonic acid is heated with sulphuric and boric acids to form the boric ester, and condensed with phenol at 10—20° to give the *compound* (I), which may be isolated by addition of acetic

$$\text{(I.)}\quad CO<\begin{matrix}C_6H(NH_2)(OH)(SO_3H)\\C_6H(NH_2)(OH)(SO_3H)\end{matrix}>C<\begin{matrix}OH\\C_6H_4\cdot OH\end{matrix}$$

acid, or by liming out and evaporation in a vacuum. This product is converted, if desired without isolation, into 4 : *8-diamino-2-p-hydroxyphenylanthrarufin-6-sulphonic acid* (*pyridine* salt described), which is a blue acid wool dye. Removal of the sulphonic group gives a *compound* which dyes chrome-mordanted wool in blue shades. Similar series of products are obtained from 4:8-dimethyldiaminoanthrarufin-2 : 6-disulphonic acid, and by using other phenols or their ethers, *e.g.*, anisole, resorcinol, quinol, naphthols, diphenyl ether, etc. C. Hollins.

Manufacture of vat dyes of the 1-thionaphthen-2'-indoleindigo series. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 274,527, 17.2.26).—Thioindoxyls containing in 4 : 6-positions halogen or alkyl groups, and hydrogen, alkyl, or halogen in position 5, are condensed with α-anils or α-halides of isatins, particularly of 5 : 7-dihalogeno-isatins (or the condensation product may be halogenated). 4 : 6-*Dichlorothioindoxyl*, m.p. 140—143°, 4 : 5 : 6-*trichlorothioindoxyl*, m.p. 150—153°, 4-*chloro*-6-*methylthioindoxyl*, m.p. 106—108°, 6-*chloro*-4-*methylthioindoxyl*, m.p. 120—123°, 4 : 6-*dichloro*-3-*methylthioindoxyl*, m.p. 135°, and 4 : 6-*dichloro*-5-*methylthioindoxyl*, m.p. 110°, are prepared, respectively, from 2 : 4-dichloro- and 2 : 3 : 4-trichloro-anilines, 5-chloro-*o*-toluidine, 3-chloro-*p*-toluidine, and 4 : 6- and 2 : 6-dichloro-*m*-toluidines, by the usual methods. The condensation of these thioindoxyls with 5 : 7-dichloro-, 5 : 7-dibromo-, and 5 : 6 : 7-trichloro-isatin α-chlorides or α-anils in chlorobenzene or xylene at 60—90° gives reddish-violet to bluish-violet vat dyes. 4 : 5 : 6-*Trichloro*-o-*toluidine*, m.p. 94—95° (*acetyl* derivative, m.p. 199—200°), is prepared from 3 : 4-dichlorotoluene by nitration, further chlorination, and reduction; 4 : 5 : 6-*trichloro*-m-*toluidine* from 4 : 5 : 6-trichlorotoluene, m.p. 41°, by nitration, separation of *m*-nitro-compound by fractional distillation, and reduction; 4 : 6-*dichloro*-3-*aminoethylbenzene* from *p*-chloroethylbenzene by chlorination, nitration, and reduction. C. Hollins.

Manufacture of azo dyes [pigments and ice-colours]. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 255,072, 7.7.26. Conv., 7.7.25).—Compounds of the type $Ar\cdot SO_2\cdot NR\cdot Ar'\cdot NH_2(o)$, in which Ar and Ar' are aromatic residues and R is alkyl or aralkyl, are diazotised and coupled in substance or on the fibre with an arylamide of 2 : 3-hydroxynaphthoic or acetoacetic acid. Benzenesulphon-*o*-aminomethylanilide gives with 2 : 3-hydroxynaphthoic *o*-toluidide or 5-chloro-*o*-toluidide a red, with bisacetoacetyltolidine a greenish-yellow dye. C. Hollins.

Manufacture of alkylated or *cyclo*alkylated arylsulphonic acids. I. G. Farbenind. A.-G. (E.P. 268,375, 25.3.27. Conv., 27.3.26. Addn. to 250,241; B., 1927, 470).—Anhydrous aromatic sulphonic acids are condensed with aliphatic or alicyclic alcohols and chlorosulphonic acid. The products show variation of the position of the sulphonic groups as compared with those obtained by the similar condensation of aromatic hydrocarbons. *E.g.*, naphthalene is sulphonated with chlorosulphonic acid, and the reaction mixture treated simultaneously with butyl alcohol and chlorosulphonic acid, and then with chlorosulphonic acid. The sodium salt of the product is a wetting and emulsifying agent.
B. Fullman.

Manufacture of *N*-monoalkyl-*p*-aminophenol. H. E. Potts. From Chem. Fabr. Grünau, Landshoff & Meyer A.-G. (E.P. 274,175, 10.4.26).—Acylated *ON*-alkyl-*p*-aminophenols (*e.g.*, *N*-methylaceto-*p*-phenetidide) are converted into *N*-monoalkyl-*p*-aminophenols by hydrolysis with sulphuric acid; the amount of the latter may be varied continuously from 6 equivalents of 50% acid (heating for 3 hrs. at 139°) to 3 equivalents of 70% ($4\frac{1}{2}$ hrs. at 146°). The acid is neutralised with chalk, and on evaporation the pure alkylaminophenol crystallises. B. Fullman.

Manufacture of anthraquinone derivatives. W. H. Perkin, A. W. Fyfe, and M. Mendoza, Assrs. to British Dyestuffs Corp., Ltd. (U.S.P. 1,636,485, 19.7.27. Appl., 5.4.26. Conv., 8.7.25).—See E.P. 252,922; B., 1926, 658.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Hygroscopic properties of cotton wool and its charred product. S. V. Gorbatschev and E. N. Vinogradova (Biochem. Z., 1927, **186**, 413—418).—The adsorption of water vapour by cotton wool and its

charred product ("granulose") has been measured at 37° in an atmosphere saturated with water vapour. The kinetics of the adsorption deviate considerably in each case from the adsorption of gases, for which Iljin has given a theoretical formula (A., 1924, ii, 92). The adsorption isotherms for cotton wool and "granulose" show that the latter possesses a distinctly greater adsorptive power at higher humidities, and is therefore to be preferred for bandages. Used clinically, "granulose" has given encouraging results. A. Wormall.

Kapok. R. O. Bishop and G. L. Teik (Malayan Agric. J., 1927, **15**, 97—103).—Samples of kapok from various sources do not differ materially in their chemical and physical properties, nor is it possible to effect a satisfactory differentiation by buoyancy tests. Extraction of kapok with fat solvents has no appreciable effect on buoyancy. The mean fibre lengths of Serdang kapok, prime Samarang, and average Samarang kapok are 6·5, 7·5, and 10·0 mm. respectively. D. J. Norman.

Determination of copper in rot-proof canvas. Bonnard and Leblanc (Ann. Chim. Analyt., 1927, [ii], **9**, 233—235).—Owing to the presence of chromium in rot-proof canvas the copper cannot be completely dissolved by acid treatment. The sample is therefore incinerated in a platinum dish and the residue fused with 5 g. of potassium hydrogen sulphate. The product is leached with hot water containing a little hydrochloric acid, and the copper in the solution is separated by a current of hydrogen sulphide. A. R. Powell.

Chemical processes for re-refining and decolorising dry-cleaners' solvent by continuous automatic methods. A. E. Flowers, F. H. McBerty, and M. A. Dietrich (Ind. Eng. Chem., 1927, **19**, 868—873).—100 kg. of goods cleaned yield 1·6 kg. of solid dirt and 1·8 kg. of dark-coloured, soluble impurity. As little as 0·1% of moisture may cause dirty solvent to mark goods subsequently treated. The odour which petroleum solvents communicate to clothes etc. is due mainly to unsaturated hydrocarbons. The centrifugal clarifier is suitable for the continuous removal of solid impurities. Distillation in steam for the removal of colour and odour is relatively costly and unsatisfactory. For this purpose a continuous process using sulphuric acid has been developed which is much superior to the employment of alkali, fuller's earth, or activated carbon. The washing apparatus is fitted with a number of discs upon which the solvent mixed with 3% of acid falls. In a second apparatus neutralisation with dilute ammonia is performed. This does not cause emulsification. The treated solvent is returned to the clarifier to remove moisture. The process is more suitable for "Stoddard-solvent" (a spirit with restricted distillation range) than ordinary petrol as the end-point of the latter is unduly raised. C. Irwin.

Measurement of the "wetness factor" of mechanical wood pulp by the Schopper-Riegler apparatus. W. Brecht and E. Schaun (Papier-Fabr. 1927, **25**, Fest- u. Ausland-heft, 45—60).—The Schopper-Riegler apparatus has attained very considerable importance for the control of the grinding operation, since in the case of ordinary "news" paper manufacture under rigorously standardised conditions, all the technically important qualities of the paper can be governed by the factor established by this test. An investigation of the sources and limits of error in the Schopper-Riegler test has led to the recommendation that the standard test should be carried out on 1000 c.c. of a suspension containing 0·30% of absolutely dry fibre at a standard temperature of 20°. The error due to variations of temperature is a fall of 0·462 "wetness" or "slowness" unit per 1° rise in temperature. Variations in the volume of the suspension with constant concentration induce a rise in the "slowness" factor with fall in volume, but not in direct proportion. The principal source of error is a variation in consistency, *i.e.*, dry fibre-substance present in the 1000 c.c. of suspension. This error is large if concentrations below 0·3% are selected for the test; on the other hand, if higher concentrations are adopted, the error is reduced but the sensitiveness of the test is much impaired. Errors in concentration may be minimised by adopting standard manipulation in preparing the suspension. It is recommended that 250 c.c. of the sample of fluid pulp be collected on a filter in a Buchner funnel and pressed to a cake of standard dry-substance by means of a brass plate and weight calculated to give a uniform pressure of 0·445 kg. per sq. cm. for 3 min. The calculated quantity of this cake necessary to give 3 g. of dry fibre is shaken with the complementary volume of water for the test-suspension. Lastly, a slight modification of the test-apparatus for eliminating the personal factor in releasing the flow for the test is recommended, or alternatively the adoption of the improved pattern of instrument devised by the Canadian manufacturers. J. F. Briggs.

Digestion of wood with liquors of low sulphite content. E. Hägglund (Papier-Fabr., 1927, **25**, Fest- u. Ausland-heft, 60—63).—In certain cases liquors containing low concentrations of sulphite are used in order to shorten the time of digestion, the proportion of sulphite being an important factor in the speed of the digestion. This is because the acidity with low concentration of sulphite is greater than with high sulphite concentration. In the first stage of the digestion, while the sulphite is combining with the lignin, there is not much difference between the two types of liquor, since this phase is not dependent on the hydrogen-ion concentration, and large quantities of lignosulphonic and sulphuric acids are not present in the liquor. In the second phase, however, the difference is very marked, but shows itself more in the larger quantity of direct cupric-reducing sugars than in the degree of digestion of the wood. The yield of cellulose is only markedly affected in the last phase of the digestion, and a liquor of low sulphite content gives in 15 hrs. the same yield of cellulose as the less acid liquor in 17 hrs. Owing to the greater hydrolytic action of the less basic liquor, the proportion of direct reducing sugars is consistently higher, even at equivalent yields of cellulose, and the quality of the cellulose is lower both in tensile strength and particularly in the folding test; the cellulose produced has a brittle fibre. J. F. Briggs.

Digestion of plant materials with nitric acid. P. Krais, K. Biltz, and O. Renner (Papier-Fabr., 1927, **25**, Fest- u. Ausland-heft, 43—44).—Several charges

on an industrial scale up to 900 kg. of wood waste, straw, and reeds have been put through with favourable results. The consumption of nitric acid has been rather higher than in the laboratory, but improvements in the recovery of gaseous products are in hand and the institution of rotary digesters of larger dimensions. The celluloses produced on the large scale have copper values of 2·4 to 2·7 and the α-cellulose content ranges between 78·2 and 86·3% according to the raw material. It is true that small quantities of hydrocyanic acid vapours are produced, but greater personal inconvenience is probably caused by the escape of nitric oxide, which at the same time acts as a warning. The yield and quality of the cellulose produced by the nitric acid process are comparable with those of sulphite pulp. J. F. BRIGGS.

Decomposition of cellulose. HEUKELEKIAN.—See XXIII.

PATENTS.

Treating plant material for the removal of incrusting materials from the fibre. F. K. FISH, JUN. (E.P. 255,029, 4.6.26. Conv., 7.7.25).—Fibrous material is fed into a digester which already contains a proportion of liquor and is provided with a device whereby liquor is continuously withdrawn from the bottom of the digester and sprayed on top of the charge. When completely charged the digester is sealed and evacuated. A further quantity of liquor is then admitted and digestion completed under pressure. During each of these operations circulation and spraying of the liquor are maintained. The cooking liquor may be acid, neutral, or alkaline, but should contain the volatile plant extractives recovered from previous operations. D. J. NORMAN.

Wadding. PAPETERIES DE LA ROBERTSAU (E.P. 265,125, 10.6.26. Conv., 29.1.26).—Non-hydrophile wadding is obtained by sizing the cellulose pulp in the hollander with, *e.g.*, 3% of resin soap using aluminium sulphate as precipitant, or with gelatin with tannic acid as precipitant. D. J. NORMAN.

Extracting fats from textiles. A. BEIL and W. A. MEYER, Assrs. to A. RECHBERG-G.M.B.H., and G. BRAUN-G.M.B.H. (U.S.P. 1,636,351, 19.7.27. Appl., 11.3.26. Conv., 18.3.25).—The material is treated with liquid methylene chloride. D. J. NORMAN.

Desizing process. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 274,795, 18.1.26).—The desizing of fibrous material of animal, vegetable, or artificial origin is facilitated by the addition of wetting agents to the desizing liquor. D. J. NORMAN.

Manufacture of artificial silk etc. W. P. DREAPER (E.P. 273,354, 11.1.26).—During the replacement of one centrifugal box by another in the centrifugal spinning process, the thread is diverted before reaching the funnel guider and collected in an untwisted condition on a reel or bobbin. The funnel is preferably of the type described in E.P. 171,719 (B., 1922, 52 A). The untwisted filaments thus obtained may conveniently be converted into staple fibre. D. J. NORMAN.

Manufacture of threads, filaments, etc. from viscose. COURTAULDS, LTD., H. J. HEGAN, and E. HAZELEY (E.P. 273,506, 19.7.26).—Hollow viscose filaments, which consist chiefly of narrow tubes practically free from spaced bubbles, are obtained by spinning a viscose solution containing 1—3% of sodium carbonate into a bath containing 9—11% of sulphuric acid, 10—14% of sodium sulphate, 8—14% of magnesium sulphate, and 0—8% of zinc sulphate, the proportions being so arranged that the bath contains a total of 25—28% of metal sulphates. D. J. NORMAN.

Manufacture of artificial materials from viscose. L. LILIENFELD (E.P. [A] 274,521 and [B] 274,690, 11.1.26). —(A) Viscose silk of high strength, *e.g.*, 2—5 and 1·5—2·5 g. per denier in the dry and wet state respectively, is obtained by spinning, preferably stretch-spinning, viscose solution into sulphuric acid of 55—85% strength. Unmatured or relatively slightly matured viscose made from alkali-cellulose that has matured only for a short time, *e.g.*, 3—36 hrs. is, in general, more satisfactory than fully ripened viscose, being less affected by the strong acid; in certain circumstances, however, it is advantageous to use a more fully ripened viscose in the preparation of which more than 40% of carbon disulphide (on the weight of cellulose) has been added. The viscosity of the spinning solution should be at least twice that of glycerin. The length of travel of the thread both in and outside the bath may vary within wide limits depending on the concentration of the acid, the ripeness of the viscose, etc. Means should be provided for diluting the acid on the thread before or during the reeling operation. Any suitable additions may be made to the spinning bath or to the viscose solution. (B) The whole or a part of the sulphuric acid in the spinning bath described above can be replaced by another strong mineral acid, *e.g.*, hydrochloric acid of 25—40% strength, nitric or arsenic acid of 60—90%, or phosphoric acid (*d* 1·5—1·86). D. J. NORMAN.

Production of foils, skins, bands, etc. from cellulose. WOLFF & CO., E. CZAPEK, and R. WEINGAND (E.P. 257,924, 27.8.26. Conv., 1.9.25).—Solutions of suitable inorganic acids or salts or mixtures thereof in organic solvents are used as coagulating agents for aqueous cellulose solutions in the manufacture of articles which have a larger cross-section than that of artificial silk. Alcohols of low b.p., particularly methyl alcohol, may be used as organic solvents, and suitable coagulating baths consist of methyl alcohol in admixture with (*a*) 40% of sulphuric acid, (*b*) 10% of magnesium chloride, or (*c*) 10% of magnesium chloride and 10% of free hydrochloric acid. Methyl alcohol alone may be used to effect a preliminary coagulation of the cellulose solution if desired. D. J. NORMAN.

Treating artificial silk produced in centrifuges. VEREIN. GLANZSTOFF-FABR. A.-G. (E.P. 265,996, 12.2.27. Conv., 13.2.26).—In the manufacture of artificial silk, particularly viscose silk, by the centrifugal process a more regular product is obtained if the freshly spun cake of silk is washed and, as far as possible, dehydrated centrifugally before removing it from the spinning pot. The silk is then reeled off and the resulting hanks are dried, preferably on a stretching carriage. To prevent damage to the cake during washing, water in a fine state of division is used. D. J. NORMAN.

Treating cellulose acetate. E. S. Farrow, jun., Assr. to Eastman Kodak Co. (U.S.P. 1,634,986, 5.7.27. Appl., 12.10.25).—Acetone-soluble cellulose acetate is produced by treating chloroform-soluble cellulose acetate, dissolved in acetic acid, with an aqueous solution of phosphoric acid. D. J. Norman.

Production of fibre half-stuff. I. G. Farbenind. A.-G. (E.P. 264,803, 9.12.26. Conv., 25.1.26).—Pulp of good quality can be obtained from flax straw, corn straw, jute, hemp, etc. by digestion under pressure with a liquor containing sodium bisulphite and sodium sulphite in the ratio 3—3·5 : 1 with addition of neutral salts, preferably those which undergo hydrolysis at elevated temperatures. A certain proportion of the alkali bisulphite and sulphite may be replaced by the corresponding alkaline-earth salts if the ratio of bisulphite to sulphite remains unaltered. D. J. Norman.

Producing white pulp of high strength. G. A. Richter, Assr. to Brown Co. (U.S.P. 1,635,637, 12.7.27. Appl., 11.7.25).—Raw cellulosic material is digested in an acid sulphite liquor in which the free sulphur dioxide is not in excess of the combined sulphur dioxide. The resulting pulp is washed, treated with a solution containing chlorine, again washed, and finally digested with a solution containing alkali. D. J. Norman.

Process of making paper from straw. C. D. Wood, Assr. to Grasselli Chem. Co. (U.S.P. 1,634,603, 5.7.27. Appl., 12.1.26).—The alkali-soluble components obtained by digesting fibrous material with soluble sulphides are incorporated with the pulp during its conversion into paper. D. J. Norman.

Treatment of [wool-scouring] liquids containing oil or fatty substances and the like. J. Avery, Assr. to Australian Lanoline Proprietary Ltd. (U.S.P. 1,638,977, 16.8.27. Appl., 27.9.23. Conv., 23.10.22).—See E.P. 205,833; B., 1925, 281.

Dry distillation of alkalised waste liquor from soda cellulose manufacture. E. L. Rinman (U.S.P. 1,638,061, 9.8.27. Appl., 4.6.24. Conv., 6.6.23).—See E.P. 217,235; B., 1925, 496.

Treating fibres etc. (E.P. 273,830, 272,165, 263,102, 274,940, 273,406, and 274,584).—See VI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Is digallic acid identical with tannin as a mordant for basic dyes? P. P. Viktorov (Z. angew. Chem., 1927, **40**, 922—925).—Synthetic *m*-digallic acid obtained by Fischer's method possesses the characteristic properties of the natural tannins, being precipitated by tartar emetic and affording precipitates with basic dyes which dissolve in excess of the acid. Digallic acid is adsorbed by cotton, the adsorption increasing with the time of immersion in the bath. With tartar emetic and basic dyes it affords dyeings on cotton which in the case of Victoria Blue B and Night Blue are deeper, purer, and bluer than the shades obtained under corresponding conditions with tannin. Methylene Blue and Malachite Green, however, afford deeper and greener shades when dyed with tannin than with digallic acid. In mordanting with tannin, the tannin is decomposed and only the digallic acid plays an active part in the formation of the antimony mordant compound, $OH\cdot Sb(C_{14}H_9O_9)_2$. In both mordant and dyeing processes the active constituent of natural tannin is that which appears in synthetic pentadigalloyl-β-glucose containing digallic acid. Doubt is thrown on the pentagalloyl-β-glucose structure assigned to Turkish tannin by Fischer, since gallic acid is only very slightly adsorbed by cotton, and in a 0·25% acetic acid bath. It is possible, however, that if Turkish tannin possesses a pentagalloylglucose structure its mordant properties are to be attributed to the 2·7—3·8% of ellagic acid also present. R. Brightman.

Patents.

Treatment of cotton fabrics containing artificial silk. Calico Printers' Assoc., Ltd., L. A. Lantz, and C. M. Keyworth (E.P. 273,830, 12.4.26).—Cotton in admixture with cellulose acetate silk can be mercerised with ordinary mercerising solutions under ordinary conditions of time, temperature, etc. without injury to the cellulose acetate if the fabric is impregnated, prior to mercerisation, with a liquid which neither mixes nor reacts with alkali solution of mercerising strength, but prevents or retards hydrolysis of the cellulose acetate. Suitable impregnating agents include benzene and its homologues, hydrogenated naphthalenes, petroleum distillates, solvent naphtha, and essential oils. D. J. Norman.

Chemically varying vegetable or artificial fibres. Heberlein & Co. A.-G. (E.P. 272,165, 29.11.26. Conv., 5.6.26).—By the use of suitable resists the process described in E.P. 255,453 and 261,792 (B., 1927, 103, 473) may be used for the production of two-colour effects on fabrics; *e.g.*, bleached cotton fabric is printed with a mixture of caustic soda and dextrin and is then drawn through a 3% solution of phosphorus oxychloride in xylene. Those parts of the fabric that have been printed with alkali show modified dyeing properties, whilst the remainder is substantially unaffected. D. J. Norman.

Preparation for sizing textile fabrics. A. F. Galvin (E.P. 254,720, 30.6.26. Conv., 4.7.25).—A sizing preparation which neither damages nor discolours textile fabrics is obtained by adding linseed oil previously boiled with a cobalt acetate, oleate, or resinate drier to a refined drying oil or light drying oil varnish, the total quantity of drier being less than 4% on the weight of drying oil present. Other constituents may be added to produce various effects. A suitable solution contains petrol 2 litres, drying oil produced as above 1 litre, tung oil 0·5 litre, soap 0·1 litre, grease 30 g., Japan wax 30 g. D. J. Norman.

Manufacture [sizing] of artificial silk and other textile fibre. Neutrasol Products Corp., Assecs. of E. Pohl (E.P. 255,909, 27.7.26. Conv., 27.7.25).—A coating material for artificial silk etc. is obtained by partially or completely saponifying a mixture of beeswax and other waxes and fatty acids, *e.g.*, 3 pts. of beeswax, 3·5 pts. of Japan wax, 3·5 pts. of stearic acid, and 1 pt. of oleic acid saponified with 1 pt. of caustic soda dissolved in 20 pts. of water. D. J. Norman.

Improving vegetable textiles. A. Meyer-Sansbœuf G.m.b.H. (E.P. 263,102, 4.11.26. Conv., 21.12.25).—Textile materials in loose or fabric form show improved

resistance to ultra-violet light, water, and dilute solutions if they are subjected to the successive action of a tanning solution containing a wetting agent, a mordanting solution, and an oiling solution containing an aldehydic or ketonic substance to control the rate of oxidation of the oil during the final drying operation; *e.g.*, cotton yarn is impregnated for 0·5 hr. at 90° in a bath containing 5% of catechu and 5% of Tetracarnit (a mixture of Turkey-red oil, pyridine, and tetrachloroethane), centrifuged, and transferred for 0·5 hr. to a 5% solution of lead acetate at 50°. The oiling bath consists of a 5% solution of linseed oil in tetrahydronaphthalene with addition of 2% of paraldehyde; after impregnation for 10 min. at the ordinary temperature, the material is centrifuged and allowed to dry.

D. J. Norman.

Treatment of cellulosic materials. W. Kershaw, F. L. Barrett, C. S. Parker, and Bleachers' Assoc., Ltd. (E.P. 274,940, 23.4.26).—Cotton fabrics are treated with sulphuric acid of parchmentising strength containing dissolved cellulose, so that, when the acid is washed out, cellulose is precipitated within and upon the fabric, giving a stiff material, which resists deformation by stretching even when wet, and, after suitable conditioning, is capable of taking embossed patterns which are not destroyed by washing or boiling. A solution of 5 pts. of pure air-dry cotton cellulose in 95 pts. of sulphuric acid (*d* 1·575—1·675) at 0° is suitable for parchmentising; it is fairly stable at low temperatures, but should not be kept many hours. D. J. Norman.

Manufacture [finishing] of fabrics. British Celanese, Ltd., W. A. Dickie, and H. Halkyard (E.P. 273,406, 1.4.26).—Embossed effects, which are not affected by ordinary laundering operations and cause no substantial weakening of the fabric, are produced on fabrics composed of or containing cellulose acetate or other thermoplastic cellulose derivatives by impregnating the fabric with a volatile liquid which causes the cellulose derivative to swell, but not to dissolve, and exposing the treated fabric to heat, *e.g.*, 110—120°, and pressure. Suitable liquids are water, aqueous solutions of, *e.g.*, ammonium thiocyanate, alcohol, etc., solutions of acetone, *cyclo*hexanone, ethyl lactate, etc. in volatile organic solvents which have no action on cellulose acetate, *e.g.*, benzene; plasticising agents and lubricants may be added if desired. D. J. Norman.

Treatment of yarns or fabrics consisting of or containing artificial silk. C. S. Parker, W. Kershaw, F. L. Barrett, and Bleachers' Assoc., Ltd. (E.P. 274,584, 29.4.26).—Crêpe-like effects are obtained on fabrics consisting of or containing cellulose esters by treating the fabric for 3—180 sec. at 0—55° with dilute nitric acid (*d* 1·15—1·25). D. J. Norman.

Apparatus for bleaching, dyeing, washing, and drying yarns or fabrics. W Winter and H. Jordan (E.P. 275,363, 31.5.26).

Arylsulphonic acids (E.P. 268,375). **Compounds of *p*-diamines and sulphur dioxide** (E.P. 269,583). —See IV.

Resinous condensation products (E.P. 274,155).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Repairing sulphuric acid chamber bottom during operation. W. C. Kendrick and M. E. Souder (Ind. Eng. Chem., 1927, **19**, 954—955).—A leak which had developed over the full width of an acid chamber bottom was repaired without stopping the chamber as follows:—A sectional wooden tunnel, covered on the outside with composition and pitch, was prepared. The chamber was put under suction, a hole cut in it, and the tunnel inserted, the sections being jointed together with acid-proof cement. The acid inside the tunnel, which had no bottom, was taken up with dry earth and the paste pushed through loose boards to form a dam outside. The repair was then carried out, the whole operation taking 25 hrs. C. Irwin.

Ammonium sulphate. Manufacture of household ammonia. G. Walmsley (Gas J., 1927, **189**, 391—392).—A considerable increase in the revenue arising from the disposal of gas-works' ammonium sulphate appears possible by the conversion of part at least into ammonia solution (10—12%) for domestic use. The process consists in distilling a concentrated solution of ammonium sulphate with lime, using a small steam-heated still of special design. Working up one ton of sulphate can be made to yield a profit of approximately £50. A. B. Manning.

Physico-chemical principles of the recovery of ammonium sulphate from ammoniacal gases and sulphuric acid. E. Terres and W. Schmidt (Gas- u. Wasserfach, 1927, **70**, 725—728, 762—766, 808—813).—The solubility of ammonium sulphate in water containing various proportions of sulphuric acid was determined at temperatures between 0° and 100°. With an acid concentration of 0—22%, the solid phase is normal ammonium sulphate, the solubility decreasing with increasing acidity at temperatures up to 40° and increasing slightly with increasing acidity at higher temperatures. With an acidity of about 22—35%, the solid phase is the double salt ammonium sulphate-bisulphate, the solubility of which increases with increasing acidity, whilst at still higher acid concentrations it is ammonium bisulphate alone, the solubility of which decreases to a minimum at an acidity of about 58%. The solubility of ammonium sulphate in water containing free ammonia decreases regularly and rapidly with increasing ammonia content, falling to zero (by extrapolation) at a free ammonia concentration of about 30% at 0°. Vapour pressure curves are also established for the above solutions and temperatures. The vapour pressure of ammoniacal solutions falls rapidly and regularly with decreasing alkalinity. With increasing acidity the vapour pressure continues to decrease, though less rapidly, the effect being more marked the higher the temperature, and breaks in the curves indicate the change in the solid phase at about 35% acidity. The bearing of these physico-chemical data on the practical manipulation of the direct and semi-direct ammonia recovery processes and of the Burkheiser ammonium sulphate-bisulphate process is discussed.

W. T. K. Braunholtz.

Manufacture of bismuth salts. F. CHEMNITIUS (Pharm. Zentr., 1927, **68**, 513—518).—The preparation of the normal and basic nitrate, oxide, hydroxide, sulphate, benzoates, subcarbonate, citrates, subgallate, subsalicylate, tartrate, lactate, oxyiodide, iodogallate, tannate, albuminate, phenoxide, and β-naphthoxide is described.

Composition of technical calcium hypochlorite and comparison of its behaviour on heating with that of bleaching powder. H. DITZ and R. MAY (Z. Elektrochem., 1927, **33**, 265—272).—Technical calcium hypochlorite (Griesheim) has been analysed and the results are compared with those obtained by Kast and Metz (cf. B., 1927, 297). The much higher value for free lime given by the latter results from their assumption that it can be regarded as the difference between the total lime and that combined as hypochlorite, no allowance being made for that present as chloride, chlorate, or carbonate. The same error is involved in their values for free lime in the bleaching powder samples examined. The results of Kast and Metz on the tendency of calcium hypochlorite and bleaching powder to decompose on heating, and on the action of carbon dioxide on bleaching powder are compared with previous results of Hoffmann and Ritter (A., 1914, ii, 612). Further experiments have been made on the behaviour of technical calcium hypochlorite when heated alone, or with addition of calcium chloride, in a current of dry air free from carbon dioxide. On the basis of the results a critical examination is made of the conclusions derived by Kast and Metz from experiments on the action of heat on technical calcium hypochlorite and bleaching powder, and on the action of carbon dioxide on bleaching powder.

H. J. T. ELLINGHAM.

Colorimetric determination of carbon monoxide by means of an ammoniacal silver solution. H. KAST and A. SCHMIDT (Gas- u. Wasserfach, 1927, **70**, 821—822).—Whereas Wein's method of determining carbon monoxide by means of palladium chloride cannot be applied to mixtures containing ethylene or acetylene, Thiele's method, using an ammoniacal silver solution, is unaffected by the presence in the mixture of up to 2% of ethylene, or of acetylene provided its concentration does not exceed one tenth of the carbon monoxide content. Methane and hydrogen do not interfere with the method, which can therefore be used for determining carbon monoxide in technical gases, *e.g.*, hydrogen for the synthesis of ammonia. W. T. K. BRAUNHOLTZ.

PATENTS.

Manufacture of sulphuric acid. C. W. FIELDING (E.P. 274,918—9, 29.1.26).—(A) Gases containing sulphur dioxide and free oxygen, with or without oxides of nitrogen and water vapour, are brought into intimate contact with nitrosylsulphuric acid or with sulphuric acid sufficiently concentrated to absorb oxides of nitrogen in a vessel consisting of a series of tubes or columns provided with mixing devices. The nitrated acid is continuously circulated and cooled, so that it acts as a true surface catalyst and oxidation proceeds in the liquid phase. (B) Intimate contact is made between the nitrosylsulphuric acid and gases containing sulphur dioxide and free oxygen by forcing the gases through the acid contained in tubes, in which are supported perforated septa above and below the surface of the liquid, or the gases are forced to pass through fine films of the acid supported on perforated or slotted sieve plates so that a foam is produced. W. G. CAREY.

Producing active silicic acid. RING GES. CHEM. UNTERNEHMUNGEN M.B.H. (E.P. 254,726, 30.6.26. Conv., 1.7.25).—Silicon fluoride is treated with small quantities of water or steam, alone or mixed with gas or other vapour, the resulting silicic acid being then washed and moulded under pressure, and the adherent moisture removed by suction without depriving the product of its hygroscopic water content. H. ROYAL-DAWSON.

Recovery of phosphorus and hydrogenated compounds thereof in the form of phosphoric acid. URBAIN CORP. (E.P. 269,908, 20.4.27. Conv., 21.4.26).—Gases generated by the combustion of phosphorus are passed into a Gaillard tower where they meet and concentrate a phosphoric acid solution which has already undergone a first concentration in a heat exchanger; the gases then proceed to a coke container in which the phosphoric anhydride is hydrated. The heat generated by the combustion of the phosphorus is used to concentrate the solution of phosphoric acid, the water evaporated being used again for hydrating. The mass of water in circulation is maintained constant.

W. G. CAREY.

Treating superphosphates. A. GAILLARD (E.P. 269,921, 22.4.27. Conv., 22.4.26).—After the first drying process the superphosphate is stored for some time in a silo to allow any unfinished chemical changes within the mass to conclude. The material is then passed through the dryer a second time. A. R. POWELL.

Decomposition of salts of complex hydrofluoric acids. A. F. MEYERHOFER (E.P. 249,860, 23.3.26. Conv., 24.3.25).—The salts are injected in powdered form by means of gas into a heated chamber, the charge being kept in continuous motion during the operation, or the powdered charge is dispersed by injecting heated neutral or active gas or steam. Substances may be introduced for reacting with the decomposing salts or for producing gases for reacting therewith, and waste gases from the decomposition chamber may be used for heating or for producing compounds required for the decomposing process. W. G. CAREY.

Oxidation of ferrous sulphate in solution. A. ELLIOTT (U.S.P. 1,636,296, 19.7.27. Appl., 31.3.26).—Air is passed through hot ferrous sulphate solution under pressure and at a temperature below 100°.

W. G. CAREY.

Manufacture of pure lead carbonate from crude lead sulphate. R. DALOZE (E.P. 273,660, 19.3.27. Conv., 3.7.26).—Crude lead sulphate, *e.g.*, the slime from lead chambers, is treated with a solution of lead acetate and excess of calcium acetate, and filtered. The calcium sulphate is separated and milk of lime is added to the solution, forming basic lead acetate, which is decomposed with carbon dioxide, precipitating the lead carbonate, and leaving lead and calcium acetates in solution to be used again. H. ROYAL-DAWSON.

Manufacture of titanium oxide. J. BLUMENFELD, Assee. of FABR. DE PROD. CHIM. DE THANN ET DE

MULHOUSE (E.P. 253,550, 11.6.26. Conv., 11.6.25).—Ilmenite ore is treated with sulphuric acid, the iron compounds are reduced to the ferrous state, and the solution is cooled to about 0° to crystallise out the ferrous sulphate. The mother-liquor is then subjected to hydrolysis and concentration for treating further quantities of ore. H. ROYAL-DAWSON.

Process of mining insoluble boron compounds. H. BLUMENBERG, JUN. (U.S.P. 1,636,455—6, 19.7.27. Appl., 23.3.27).—(A) Hot calcium chloride solution is introduced into the calcium-boron deposit, thus disintegrating and dissolving a portion; the resulting magma is removed and cooled, whereby the dissolved compounds are precipitated. These are removed, leaving the calcium chloride solution with only a small content of calcium-boron compounds. (B) Hot ammonium chloride solution may be used in the same manner. W. G. CAREY.

Conversion of oxides into anhydrous fused chlorides. I. G. FARBENIND. A.-G., and K. STAIB (E.P. 275,116, 5.3.27).—Finely-divided oxides, *e.g.*, oxides of calcium, strontium, magnesium, zinc, are introduced into a heated reaction chamber with the aid of and suspended in a mixture of chlorine and carbon monoxide, or carbon is mixed with the oxide and chlorine alone is used to introduce the oxide. W. G. CAREY.

Production of alkali-metal sulphydrates. E. E. NAEF, Assr. to TUBIZE ARTIFICIAL SILK CO. OF AMERICA (U.S.P. 1,636,106, 19.7.27. Appl., 26.1.24. Conv., 31.1.23).—See E.P. 214,358; B., 1924, 511.

Production of cobaltous acetate. H. SCHATZ, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,637,281, 26.7.27. Appl., 28.10.26. Conv., 27.11.25).—See E.P. 262,075; B., 1927, 601.

Dissolving a mixture of hafnium and zirconium phosphates and separating hafnium and zirconium. A. E. VAN ARKEL and J. H. DE BOER, Assrs. to N.V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,636,493, 19.7.27. Appl., 4.1.26. Conv., 6.6.24).—See E.P. 235,217; B., 1925, 713.

Production of activated nitrogen and of oxides of nitrogen therefrom. W. A. BONE (U.S.P. 1,639,584, 16.8.27. Appl., 19.2.24. Conv., 28.2.23).—See E.P. 217,277; B., 1924, 712.

Drying agent (E.P. 272,173).—See I.

Carbon monoxide and hydrogen (E.P. 274,610).—See II.

VIII.—GLASS; CERAMICS.

Determination of the constitution of glasses. K. TABATA (J. Amer. Ceram. Soc., 1926, **9**, 823—849).—The method used is based on experiments on the causes of devitrification of glasses. Devitrification is arbitrarily defined in five degrees, which are illustrated by photographs. The degree of devitrification varies directly with the amount of silica dissolved in the glass. The experiments were conducted on alkali silicates and on alkali lead silicates. The constitution of the former is $xNa_2O,2SiO_2+yK_2O,2SiO_2+zSiO_2$, and of ordinary flint glasses is $x(R_2O,2SiO_2,PbO,2SiO_2)+y(R_2O,2SiO_2)+z(PbO,2SiO_2)+wSiO_2$. The dissolving powers of $Na_2O,2SiO_2$ and of $K_2O,2SiO_2$ for silica vary considerably, owing to the difference in the atomic structure of both atoms of sodium and potassium. In accordance with the constitution of glasses thus determined, glass-melting pots should be rich in either alumina or silica according to the solvent power of the glass for silica. F. SALT.

Resistance to abrasion of ceramic glazes: its control and methods of determination. A. C. HARRISON (J. Amer. Ceram. Soc., 1927, **10**, 77—89).—The resistance to abrasion of ten brands of hotel ware was tested by allowing silica sand to fall through a funnel orifice 0·4 in. in length and 0·24 in. in width from a height of 40 in. on to the glazed surface, and measuring the degree of abrasion by means of a glarimeter. The resistance of the glazes was proportional to the percentage relative humidity of the atmosphere. A high degree of accuracy was obtained by this method when the humidity was maintained constant. A simpler method, suitable for works control, is described, in which abraded samples are referred to arbitrary standards. The effect on the resistance to abrasion of varying the silica and alumina content, the glaze thickness, and the firing treatment was studied on a number of experimental glazes. Insufficient data were obtained to indicate definitely the effect of varying the silica and alumina contents, but the resistance was inversely proportional to the thickness, and was markedly increased by a higher glost fire. The type of body used was of secondary importance, the glaze composition and the temperature of the glost fire being the chief factors. F. SALT.

Chromium red glaze. C. F. BINNS and E. CRAIG (J. Amer. Ceram. Soc., 1927, **10**, 73—74).—The red colour of basic lead chromate has been successfully utilised in producing a red in- or under-glaze colour from a batch mixing of 200·60 pts. of red lead, 14·40 pts. of potassium dichromate, 6·12 pts. of soda ash, 25·80 pts. of clay, and 54·00 pts. of flint. The whole glaze was fritted, ground, and fired at cone 08. F. SALT.

Gas-fired enamelling furnaces for cast iron. H. L. READ (J. Amer. Ceram. Soc., 1926, **9**, 797—804).—The relative efficiencies of gas-fired and electrically-heated enamel kilns are compared. Details of the construction are given, together with operating data, of a kiln of the surface-combustion type. F. SALT.

Defects in enamel due to cast iron. A. MALINOVSZKY (J. Amer. Ceram. Soc., 1926, **9**, 805—813).—To determine the cause of pinholding and blistering of enamel on cast iron, records were kept of changes in materials, methods, etc. Samples of castings were examined microscopically, and photomicrographs indicate that pinholing and blistering are due almost entirely to the condition of the carbon in the iron, and not merely to the presence of carbon or of other ingredients. F. SALT.

Coloured [ceramic] bodies. C. F. BINNS and H. E. WARDNER (J. Amer. Ceram. Soc., 1927, **10**, 75—76).—Experiments with different mixtures of raw materials led to the preparation of blue, green, red, brown, and yellow bodies of uniform shrinkage and porosity, which could be worked together successfully, and a suitable colourless, transparent glaze maturing at cone 04 was

evolved. The blue and green body mixtures were also used successfully as engobes on a body made up of the other three. F. Salt.

Impact and static transverse strength [modulus of rupture] of wet-process electrical porcelain. L. Navias (J. Amer. Ceram. Soc., 1927, **10**, 90—97; cf. B., 1926, 823).—Tests were made with a Charpy (pendulum) impact machine, adapted for testing porcelain specimens varying from ½ to 1⅛ in. in diameter, the support for the test pieces being variable. Results are given for cylindrical specimens of wet-process porcelain 6 in. long, tested across a 4-in. span. Specimens of ¾, 1, and 1⅛ in. diameter gave an average value of 1·53 ft.-lb./sq. in., with an average deviation of 2·4%. Similar specimens were tested for modulus of rupture across a 4-in. and a 5-in. span; an average value of 11,000 lb./sq. in. was obtained in each case. F. Salt.

Gas-expansion porosimeters [for ceramic products]. A. E. MacGee (J. Amer. Ceram. Soc., 1926, **9**, 814—822).—Three types of porosimeters based on the principle of gas expansion are described, which are modifications of the original Washburn–Bunting type (cf. B., 1922, 253 a). A direct-reading porosimeter enables both the porosity and the apparent sp. gr. to be read directly from burettes; another, of the indirect-reading type gives accurate results with test pieces varying widely in shape and size; and, finally, an instrument which operates without the use of a vacuum pump, by means of which the porosity of full-sized bricks can be determined.

Laboratory apparatus for the refining of Pacific Northwest kaolin. R. J. Clark (J. Amer. Ceram. Soc., 1927, **10**, 98—108).—The various methods of purifying clay are briefly reviewed. Samples of kaolin from Washington State were washed in a laboratory plant consisting of a feeding hopper, a rotary blunger, a sand-settling tank, a cone classifier, and two large settling tanks. A small quantity of alkali was added during blunging. The minimum feeding rate of the hopper was 240 lb. of crushed kaolin per hour. From 30 to 38% of the crude kaolin consisted of china clay, the impurities being quartz, muscovite, partially decomposed felspar, and a small amount of ferruginous matter. F. Salt.

Refractories for oil-gas manufacture. H. J. Knollman (J. Amer. Ceram. Soc., 1926, **9**, 860—887).—In generators of the two-shell type used for the manufacture of gas from crude oil, the refractories have to withstand alternating oxidising and reducing conditions, the latter strongly predominating. Other destructive factors are the deposition of carbon in the bricks, abrasion by hot gases, effect of long flames, abrupt changes of temperature, action of salt water, sulphur, and supersaturated steam. Bricks in the lower part of the generators must be hard-fired and highly refractory; those in the upper part must have a dense surface to resist the action of steam, hot gases, etc. In addition to fireclay bricks, other suitable materials are magnesite, silica, carborundum, fused magnesia, alumina, and spinel. The thermal properties of these materials are discussed. F. Salt.

Properties of refractories in the metallurgy of zinc. I. Raw materials and body mixtures now in use. E. S. Wheeler and A. H. Kuechler (J. Amer. Ceram. Soc., 1927, **10**, 109—131).—The results of a preliminary investigation of the properties of the raw materials used in making zinc retorts are reported. Confirmation of the following points was obtained: the properties of the clays varied but slightly; the use of old retort material as grog in making new retorts must be carried out with care; it must be free from slag and made from clay of good quality; a portion of the grog should consist of calcined flint clay of good quality; the presence of zinc lowers considerably the deformation points of fireclays and body mixtures. F. Salt.

Pickling iron for enamelling purposes. Staley. —See X.

Patents.

Furnace for melting and refining glass etc. J. S. Shaw (U.S.P. 1,636,151, 19.7.27. Appl., 16.6.24. Conv., 23.8.23).—See E.P. 224,622; B., 1925, 100.

Drawing of pieces of silica glass or any other vitreous material. Quartz & Silice (E.P. 270,692, 29.4.27. Conv., 4.5.26).

IX.—BUILDING MATERIALS.

Preliminary petrographic study of Portland cement. J. L. Gillson and E. C. Warren (J. Amer. Ceram. Soc., 1926, **9**, 783—786).—A petrographic study was made of five common brands of Portland cement clinker. In each case more than 95% of the material consisted of three crystalline constituents, two of which greatly predominated. The optical properties of these three constituents were determined and were found not to agree with any of the compounds of the $CaO–Al_2O_3–SiO_2$ system produced artificially by Rankin and Wright. One of the three crystalline compounds, which is biaxial, optically positive, and has indices of refraction of 1·718, 1·720, and 1·733, was also found in the set concrete, proving it to be inert and unaffected by water, a fact which was confirmed by *X*-ray examination. F. Salt.

Wood preservation. Production of acid by wood-rotting fungi. L. P. Curtin (Ind. Eng. Chem., 1927, **19**, 878—881).—The difficulty in finding a satisfactory inorganic wood preservative lies in the fact that toxicity implies solubility and that soluble salts cannot be very permanent. If, however, a sparingly soluble salt can be dissolved by some secretion of the fungus, the dilemma may be avoided. Tests made in nutrient jelly with *Fomes annosus* and other fungi and also moulds, with suitable indicators, showed a development of acid. Sodium alizarinsulphonate was the most suitable indicator. The result was confirmed with wood which had been neutralised and sterilised. Calcium and strontium carbonates precipitated in agar-syrup gels were dissolved by acids produced in this way. The acid solution is of approximately p_H 5. C. Irwin.

Patents.

Manufacture of artificial stone. M. Harnisch (E.P. 271,108, 13.5.27. Conv., 14.5.26).—Residue or waste of asbestos material, *e.g.*, serpentine, is treated

with dilute sulphuric acid (*d* 1·02—1·04), and kept for some time; it is then mixed with a binder, *e.g.*, sodium silicate and calcium chloride, or tar, forming a plastic mass, which is moulded to shape and dried in the air.

H. Royal-Dawson.

Production of heat-insulating material. H. and J. Scheidemandel (E.P. 253,919, 17.6.26. Conv., 18.6.25).—A hot solution of aluminium sulphate or zinc sulphate is stirred into a hot aqueous suspension of calcium or barium hydroxide, to which asbestos, magnesia, or kieselguhr may be added. The viscous mixture is poured into moulds to harden, and then dried.

H. Royal-Dawson.

Production of a cold glaze upon building material, such as concrete, sandstone, clay, brickwork, etc. K. Friedrich (U.S.P. 1,636,860, 26.7.27. Appl., 8.10.21). —See E.P. 190,267; B., 1923, 146 A.

Wood preservation. A. M. Howald, Assr. to Grasselli Chemical Co. (U.S.P. 1,638,440, 9.8.27. Appl., 23.1.24).—See E.P. 228,119; B., 1925, 284.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Proposed theory of the hardening and tempering of steels. O. E. Harder and R. L. Dowdell (Trans. Amer. Soc. Steel Treat., 1927, **12**, 51—68).—The mechanism of recrystallisation from γ- to α-iron on cooling is discussed. When the rate is slower than that required to produce maximum hardness, there is a slight coalescence of the carbide, the particles of which are not stable enough to resist acid reagents and the specimen etches dark, giving troostitic microstructure. Martensite needles follow the octahedral planes in austenite, which are those of greatest atomic concentration and interplanar distance, and therefore those of easy slip. The austenite previous to recrystallisation has been stressed sufficiently to cause slip. After quenching in oil, the stresses are insufficient to produce slip and more austenite is retained. The hardness of martensite may be due to intra-atomic forces, the extremely fine α grain-size, the saturation of the α lattice with carbon atoms, the distortion of the lattice caused by stress, and the interference to slip caused by the martensite needles. The evolution of heat in a quenched steel may be due to the shrinkage of the expanded space lattice.

T. H. Burnham.

Effect of silicon, nickel, chromium, and tungsten on the hardening of tool steel. W. Haufe (Stahl u. Eisen, 1927, **47**, 1365—1373).—The number of annealings at temperatures between 780° and 1030° followed by quenchings in water at 0° before hardness cracks begin to appear in a hypereutectoidal steel (1·2% C) to which varying amounts of silicon, manganese, nickel, chromium, and tungsten have been added, has been determined, and the results are reproduced in graphical form, together with the corresponding volume changes. With increasing silicon content, the tendency to fracture on repeated hardening is increased at the higher temperatures, but high-silicon steels may be annealed up to 930° and quenched in oil without showing hardness cracks. Manganese decreases the hardening range of hypereutectoidal steels very considerably, so that, by hardening at the usual temperatures, there is a tendency towards the production of cracks after quenching. Hypereutectoidal steels with a sufficiently high manganese and silicon content behave like plain carbon steels when hardened at 780°, and better than the plain steel when hardened at 830°. Up to 1% Cr decreases the tendency of high carbon steels to crack during hardening; with 1—2% Cr these steels become extremely hard without, however, losing their toughness, and hardness cracks may be avoided by quenching from 930° in oil. Nickel has scarcely any action on the hardening properties of hypereutectoidal steel, and the action of tungsten is uncertain owing to the separation of carbide during annealing.

A. R. Powell.

Effect of heat treatment on the combined carbon in grey cast iron. E. L. Roth (Trans. Amer. Soc. Steel Treat., 1927, **12**, 27—40).—Samples from two grey cast irons $\frac{5}{32}$ in. thick, containing 3·52 and 3·25% of total carbon, respectively, when heated at temperatures from 625° to 925° for $\frac{1}{2}$—3 hrs., showed that the combined carbon reached a maximum at 760°. At this temperature very little increase in precipitated graphitic carbon was obtained by heating longer than $\frac{1}{2}$ hr., but the same amount of graphitisation was obtained by heating for longer periods at lower temperatures. Silicon did not have a marked effect on graphitisation. Microscopic examination showed that the formation of graphitic carbon was not propagated from the edges to the centre. Little change occurred in the amount of total carbon. It is considered that, on heating, the Fe_3C in the matrix begins to decompose into iron and graphite, and up to 760° the iron can only dissolve graphite to a limited extent, but is capable of dissolving the graphite above this temperature, so that a minimum amount of Fe_3C is found at this point.

T. H. Burnham.

Theory of pickling of sheet iron and steel for enamelling purposes. H. F. Staley (J. Amer. Ceram. Soc., 1926, **9**, 787—796).—Efficient pickling depends on rapid dissolution, with copious evolution of hydrogen, of a thin layer of iron immediately beneath the scale. The rate of pickling depends upon the amount of acid present, the proportion thereof which is ionised, and the mobility of the ions. The most effective concentrations of acids lie between 15% and 20%. The viscosity of acid solutions is decreased, and the particles are rendered more mobile by heating. Hydrochloric acid is more effective than sulphuric; it acts more rapidly, with dissolution of less iron. The addition of small amounts of sodium chloride has a marked effect upon the pickling action of sulphuric acid. The chief object of adding auxiliary substances is to reduce the amount of acid fumes from pickling vats, and thus permit the use of highly efficient concentrations of acid. Hydrogen absorbed by metals may be removed by immersing the metals in boiling water for a few minutes.

F. Salt.

Analysis of iron and steel. Determination of silicon, phosphorus, sulphur, and manganese. M. Marqueyrol and L. Toquet (Ann. Chim. analyt., 1927, [ii], 9, 225—233).—10 g. of the sample are dissolved in 80 c.c. of nitric acid (*d* 1·33), to which are added, in small quantities at a time, 5 g. of potassium chlorate. The solution is evaporated to dryness and the nitrates

are converted into chlorides by evaporation with hydrochloric acid. The silica is removed by filtration and determined as usual; the filtrate is diluted to 500 c.c. in a graduated flask, and 250 c.c. are used for the determination of sulphur, 150 c.c. for manganese, and the remainder (measured accurately) for phosphorus. The last portion is heated to 70—80°, treated with 60 c.c. of molybdate mixture, and just neutralised with ammonia. The yellow precipitate is collected, washed, and dissolved in ammonia, the solution treated with 10 c.c. of 35% ammonium nitrate, 2 c.c. of nitric acid, and 5 c.c. of molybdate mixture, and the resultant yellow precipitate collected, washed, dried, weighed, and calculated to phosphorus. For the determination of sulphur, 20 c.c. of 12% barium chloride solution are added to the solution, which is then boiled, set aside for 12 hrs., and filtered, the precipitate being washed with 10% hydrochloric acid containing 1% of barium chloride, then with water, dried, ignited, and weighed. Manganese is determined by converting the chloride solution back into nitrate by repeated evaporation with nitric acid, precipitating the manganese as dioxide by boiling with 2 g. of potassium chlorate, and dissolving the precipitate in an excess of standard hydrogen peroxide. A. R. POWELL.

Iron–carbon–vanadium alloy for Brinell balls. G. W. QUICK and L. JORDAN (Trans. Amer. Soc. Steel Treat., 1927, **12**, 3—26).—10 mm. balls of iron–carbon–vanadium alloy containing 2·93% C and 13·5% V, quenched from 850° in water, tempered at 100°, and work-hardened were used in testing hardened steels in comparison with ordinary Brinell balls and worked-hardened and etched Hultgren balls. The flattening of the alloy balls was least and the ordinary balls showed the greatest deformation. For testing steels up to 700 Brinell hardness, however, the alloy balls offered no appreciable advantage over Hultgren balls. When testing hard steels, balls deform both elastically and plastically, the former type of deformation playing an important part. For very hard materials, therefore, a ball with greater resistance to elastic deformation such as corundum or diamond is necessary. T. H. BURNHAM.

Aluminium–silicon alloy "alpax," and its applications. L. GUILLET, DE FLEURY, and S. DE LAVAUD (Compt. rend., 1927, **185**, 327—329).—Alpax is a light alloy of silicon and aluminium in eutectic proportions (13% Si), which is used for making moulded articles on account of its small shrinkage after quenching. The microscope shows a fine structure of silicon grains in aluminium, but annealing even at low temperatures produces coalescence and impairs its mechanical properties. Alpax may be refined before pouring by means of the alkali fluorides and chlorides, and its mechanical properties then depend on the temperature of pouring, the rate of cooling, and the amount of sodium present. J. GRANT.

Influence of sulphur on copper. P. SIEBE (Z. Metallk., 1927, **19**, 311—315).—The presence of sulphur in copper leads to the formation of sulphur dioxide during melting and casting, whereby the castings rise in the mould and are extremely porous. If the sulphur is present as cuprous sulphide and oxidising conditions are carefully avoided during melting, sulphur has a much less deleterious effect on the mechanical properties of the metal than has oxygen owing to the greater softness and plasticity of cuprous sulphide compared with cuprous oxide. Up to 0·12% S has very little effect on the mechanical properties of copper: larger proportions of sulphur increase the hardness and tensile strength very slightly but appreciably reduce the resistance to impact and the ductility. The rate of change of the values for the various mechanical properties is, however, much less for increasing amounts of copper sulphide than for the corresponding quantities of cuprous oxide. A. R. POWELL.

Heat treatment and structure of $\alpha + \beta$-brass. R. HINZMANN (Z. Metallk., 1927, **19**, 297—303).—The effect on the structure of an $\alpha + \beta$-brass (50% Cu, 2% Pb, 48% Zn) of extrusion at 720° followed by annealing at various temperatures has been investigated. As the extension was effected just within the β range, and during passage through the press the metal cooled below this range into the field of duplex structure, the microstructure varied from one end to the other of the pressed rod. The first portion passing through the press completely as β showed on cooling evenly distributed needle-like crystals of β in a ground-mass of α, the middle portion passing through the press at about the decomposition point of β showed some of the β needles to have been broken up or elongated in a longitudinal direction, and the end portion of the rod which had been extruded below the decomposition point of β had a granular structure composed of evenly distributed fragments of β needles in a ground-mass of α. The mechanical properties of the end portion of the rod were therefore less satisfactory than those of the first portion, and this defect could not be completely eliminated by annealing again at 750—800° as the middle portion had a higher recrystallisation temperature and formed coarser crystals than the end portion. Characteristic photomicrographs of the different structures obtained are given. A. R. POWELL.

Separation of vanadium from tungsten. S. G. CLARKE (Analyst, 1927, **52**, 466—467).—In the presence of large and moderate amounts of tungsten good results for vanadium may be obtained by adding to the solution 10 c.c. of hydrofluoric acid, neutralising with ammonia, adding 20 c.c. of hydrochloric acid, diluting to about 300 c.c., and precipitating with cupferron (1 g. in about 20 c.c. of water). The precipitate is filtered off after 15 min. on a pulp filter, washed with dilute cupferron solution containing a few drops of 1 : 3 sulphuric acid, and burnt off in a platinum dish. If the vanadium pentoxide can be kept unfused it may be dissolved in concentrated sulphuric acid, diluted to 200 c.c., and after adding 20 c.c. of saturated sulphur dioxide solution, boiled for 20 min. and titrated with $0{\cdot}1N$-permanganate at 80°. If the oxide is fused it is brought into solution with fusion mixture. Impure tungstic oxide, as separated from steel, may be dissolved in sodium hydroxide or ammonia and boiled, and the remaining undissolved ferric hydroxide collected on the filter, washed, dissolved in hydrochloric acid, and added to the main steel solution. The vanadium is then recovered as above.

D. G. HEWER.

Electrolytic deposition of chromium. F. Salzer (Z. Elektrochem., 1927, 33, 309).—Contrary to the statement by Liebreich (cf. A., 1927, 322), the author's chromium plating process is used commercially.
H. J. T. Ellingham.

Economic and social development of the American iron and steel industry. T. W. Robinson (Iron and Steel Inst., Sept., 1927. Advance copy. 19 pp.).

Refractories in metallurgy of zinc. Wheeler and Kuechler.—See VIII.

Patents.

Protecting iron or steel melts in furnaces or converters from oxidation and adsorption of gases. B. Zwiebel (E.P. 275,002, 28.6.26).—A reducing atmosphere is formed over the surface of the slag of an iron or steel melt by introducing a heavy hydrocarbon, *e.g.*, coal tar, in either a solid or atomised condition, into the furnace at an appropriate time towards the end of the process. Adaptations suitable for Siemens-Marten furnaces and converters are described. C. A. King.

Making malleable iron castings. H. A. Schwartz, Assr. to Nat. Malleable & Steel Castings Co. (U.S.P. 1,636,657, 19.7.27. Appl., 7.12.26).—In making malleable castings of low silicon content, a compound which tends to increase graphitisation is added to the iron, and the metal is cast with minimum separation of carbon. The casting is afterwards annealed to cause precipitation of finely-divided carbon. C. A. King.

Manufacture of moulded castings of non-rusting chromium-nickel steel. F. Krupp A.-G. (E.P. 266,305, 22.1.27. Conv., 18.2.26).—For making small castings of a nickel-chromium steel fragments of the steel are charged into a hopper above the mould, covered with a deoxidising flux, and melted down by means of an oxy-hydrogen flame in which a large excess of hydrogen is used. As soon as the metal is melted the mould is rotated rapidly so as to force the molten metal into all the contours of the mould. A. R. Powell.

Purifying iron scale. A. Lucas (E.P. 275,099, 14.2.27).—For the production of iron oxide suitable for use in alumino-thermic mixtures, mill scale is crushed between rollers which are not under sufficient pressure to crush impurities harder than the scale. The crushed scale is then screened or treated with an air blast.
C. A. King.

Selective flotation of minerals from crude ores. O. Wiser (U.S.P. 1,636,974, 26.7.27. Appl., 21.6.26).—Ore pulp containing lead, zinc, and iron sulphides is treated with sulphur dioxide (0·05—1·50 lb. of sulphur per ton of ore), and lead sulphide is floated preferentially from an approximately neutral pulp. C. A. King.

Treatment of ores, wastes, oxides, metals, and other substances. M. Fourment (E.P. 265,548, 3.12.26. Conv., 2.2.26).—Metallic values are obtained by volatilisation by means of high-frequency currents induced by a winding not containing iron. Secondary crucibles may be used for fractional condensing purposes. A convenient apparatus consists of a deep cylindrical crucible into the bottom of which the raw material is fed by a central vertical tube. In the annular space formed landing stages may be provided with run-off pipes for the separation of volatilised components. The degree of fractional distillation is regulated by separately winding the different vertical stages of the column or crucible. C. A. King.

Sintering refractory materials [*e.g.*, zirconium]. N. R. Davis, and Metropolitan-Vickers Electrical Co., Ltd. (E.P. 274,283, 26.7.26).—The powdered refractory metal, *e.g.*, zirconium, is pressed into a rod and heated *in vacuo* in a wire-wound resistance furnace at 350° to drive off occluded gases at a temperature below that at which they combine with the metal. The temperature is then raised to 800—1000° and heating continued until all volatile matter is expelled. After cooling in the vacuum the partly sintered mass is transferred to another furnace in which it is heated to the sintering temperature by passing a current through it or by induction. The final heating may be conducted in a vacuum or in an atmosphere of an inert gas either at the ordinary or at a reduced pressure. A. R. Powell.

Metallurgical furnaces. Carborundum Co., Ltd., Assees. of C. E. Hawke (E.P. 257,261, 16.8.26. Conv., 20.8.25).—The refractory lining is composed of a material, such as silicon carbide, many times as heat-conductive as fireclay, and is made of a thickness such that heat is conducted through it at a rate which ensures the formation upon it of a congealed protective layer of the fused furnace contents. The lining may be built up of blocks shaped to provide passages for the circulation of a cooling medium (air). To conserve the heat conducted through the lining the circulated air is subsequently supplied to the tuyères or otherwise utilised. H. Holmes.

Furnace for treating refractory ores containing precious metals. G. W. B. Evans, Assr. to Evans Ore Reduction Co. (U.S.P. 1,636,467, 19.7.27. Appl., 13.8.26).—Ores are treated in an atmosphere of hydrogen in a rotating cylindrical chamber which is enclosed in an outer chamber also filled with hydrogen.
C. A. King.

Ore furnace. F. M. Simonds, Assr. to A. F. Hyde (U.S.P. 1,637,163, 26.7.27. Appl., 19.2.25).—An ore-treating furnace consists of a number of superimposed distributing chambers having perforated walls. A second perforated ore-confining wall surrounds the distributing chambers to form a space for descending ore. Different qualities of gas may be supplied to the different chambers of the furnace. C. A. King.

Metallic composition. H. Boving, Assr. to Western Electric Co., Inc. (U.S.P. 1,636,763, 26.7.27. Appl., 26.5.20).—When making an alloy containing metals having a low volatilisation temperature, the finely-divided components are mixed and melted under sufficient pressure of an inert gas to inhibit volatilisation of the volatile components. C. A. King.

Fluxes for soldering. J. Blackford (E.P. 274,693, 16.11.26).—A flux for use with iron, brass, etc. consists of anhydrous zinc chloride 1 pt., finely ground ammonium chloride 8 pts., and sodium fluoride $1\frac{1}{4}$ pts.
H. Royal-Dawson.

Welding copper and aluminium. J. V. Capicotto, Assr. to Dubilier Condenser Corp. (U.S.P. 1,636,656, 19.7.27. Appl., 10.6.24).—Aluminium is brought into

contact with copper coated with solder and the junction spot-welded. C. A. KING.

Steel of high-temperature stability. W. R. SHIMER, Assr. to BETHLEHEM STEEL Co. (Re-issue 16,683, 19.7.27, of U.S.P. 1,624,075, 12.4.27).—See B., 1927, 415.

Furnace for heating metal sheets etc. BRITISH FURNACES, LTD., and E. W. SMITH (E.P. 275,513, 23.3.27).

Fuel [for welding etc.] (U.S.P. 1,637,187).—See II.

Coating metals (E.P. 274,333 and 274,355).—See XIII.

XI.—ELECTROTECHNICS.

Strength of electrical porcelain. NAVIAS.—See VIII.

Chromium. SALZER.—See X.

PATENTS.

Oxide cathodes. H. WADE. From N. V. PHILIPS' GLOEILAMPENFABRIEKEN (E.P. 274,981, 6.5.26).—A metal wire, *e.g.*, of platinum or platinum alloy, preferably coated with an alkaline-earth oxide, is wound helically on a core of highly refractory metal, preferably a poor conductor of heat, *e.g.*, tungsten or molybdenum. J. S. G. THOMAS.

Electrical heating-body for high temperatures especially for ceramic, metallurgical, and chemical processes. F. WECKERLE, Assr. to STUDIENGES. FÜR WIRTSCHAFT U. IND.M.B.H. (U.S.P. 1,637,167, 26.7.27. Appl., 24.8.25. Conv., 28.8.24).—See E.P. 239,222; B., 1925, 927.

[Cooling device for use when] coating articles with metal in vacuum by electrode dispersion. W. A. F. PFANHAUSER (E.P. 275,523, 14.4.27).

Apparatus for analysis (E.P. 252,207).—See I.

Conversion of heavy hydrocarbon oils into lighter oils (E.P. 275,120).—See II.

XII.—FATS; OILS; WAXES.

Trichloroethylene as a solvent in the determination of fats. J. GROSSFELD (Chem.-Ztg., 1927, **51**, 617—618).—The advantages of trichloroethylene as compared with ether are enumerated, and a formula is given for determining the quantity of fat in a simple mixture by direct extraction with the solvent and weighing the residue left after evaporation of the solvent from an aliquot proportion of the solution. S. I. LEVY.

Experiments on olive oil at Ghaba. M. ROUSSEAU (Bull. Mat. Grasses, 1927, 223—231).—During 1925—1926 experiments were conducted by the Department of Agriculture of Tunis to test the effect of decantation by centrifuges on the quality and preservation of olive oil. Samples of oil separated from the pulp by the older decantation method of ladling, by the Hignette, Sharples, and Alfa-Laval supercentrifuges respectively, were examined both before and after filtration in respect of acidity, taste, and power of keeping. The results prove that the centrifugal separation of the oil does not provoke any change in the quality of the resultant products, and that the keeping quality and taste of the oil are not less inferior to those of oil produced by the older methods of separation. The results for oil separated by decantation by ladling, by the Hignette centrifugal filter, and by the Sharples and Alfa-Laval supercentrifuges respectively were: acidity 0·5, 0·5, 0·45, 0·45%; water, 1·7, 1·6, 0·36, 0·40%; mineral matter and other impurities, 0·32, 0·18, 0·04, 0·04%. H. M. LANGTON.

Action of heat and blowing on linseed and perilla oils and glycerides derived from them. J. S. LONG, W. S. EGGE, and P. C. WETTERAU (Ind. Eng. Chem., 1927, **19**, 903—906).—The synthesis of linolenic monoglyceride from linseed and perilla oils is described, the products being fairly pure and showing association in benzene. Very little difference exists between the glyceride obtained from linseed oil and that from perilla oil. On heating at 225° linolenic monoglyceride apparently changes to an isomeride which does not form a solid hexabromide. At 250°, however, this isomeric change is masked by changes producing rapid increase in mol. wt. and decrease in iodine value. The glyceride rapidly decomposes at 293°. The reactions produced by blowing and by heating oils are discussed and contrasted. S. S. WOOLF.

Effect of foreign oleaginous seeds, when crushed with flax seed, on the drying and bodying properties of linseed oil. W. H. EASTMAN and W. L. TAYLOR (Ind. Eng. Chem., 1927, **19**, 896—897).—Raw linseed oil pressed from flax seed containing 6% of dockage (rich in small oleaginous seeds) was compared with oil pressed from the same seed after removal of all the foreign seeds by means of suitable cleaning machinery. Differences in the constants, particularly the iodine value and n_D^{25}, are noticed in the raw oils, and are maintained or accentuated when the oils are boiled, blown, heat-bodied, bleached, refrigerated, etc. S. S. WOOLF.

Chaulmoogra-group oils. II. Refining and isolation of hydnocarpic acid. G. A. PERKINS, A. O. CRUZ, and M. O. REYES (Ind. Eng. Chem., 1927, **19**, 939—942).—*Hydnocarpus wightiana* oil is preferable to chaulmoogra oil both as to cost and quality, and in a refined state is very suitable for use in the treatment of leprosy. Details of the refining of the oil, chiefly to remove free fatty acids, are given. The preparation of hydnocarpic acid in unprecedented yield (150 g. of esters per kg. of oil) and quality from *H. wightiana* oil is described, nearly pure ethyl hydnocarpate being obtained by fractionating the ethyl esters of the oil. Characteristics of some minor oils of this group are also tabulated. S. S. WOOLF.

Determination of some physical constants of marine animal oils. H. MARCELET (Compt. rend., 1927, **185**, 455—457).—The heats of combustion, ignition temperatures, densities, fluidities, and viscosities of twenty-nine marine animal and fish oils have been determined. C. W. GIBBY.

Detection of whale oil. M. TSUJIMOTO (Chem. Umschau, 1927, **34**, 217).—A reply to Davidsohn's criticism (B., 1927, 303) of the author's suggested method of detection of whale oil (B., 1926, 986), pointing out probable causes of the discrepancies. E. HOLMES.

Origin of the yellow colour of beeswax. G. F. JAUBERT (Compt. rend., 1927, **185**, 405—406). In view

of the predilection of bees for mignonette, yellow-weed (*R. luteola*), heather, parsley, etc., it is probable that the 1 : 3-dihydroxyflavone, m.p. 285°, previously observed in beeswax (B., 1927, 707) is accompanied by traces of other derivatives of this dye (cf. Rivière and Bailhache, Ann. Sci. agron., 1920, **38**, 82). R. Brightman.

Marine-animal and fish oils for motors. Lumet and Marcelet.—See II.

Action of cathode rays on drying oils. Long and Moore.—See XIII.

Oil in soya beans. Cole and others.—See XVI.

Cacao butter substitutes. Knapp and others.—See XIX.

Patents.

Hydrolysis or saponification of glycerides or other organic esters. V. R. Kokatnur (E.P. 251,290, 23.4.26).—Glycerides or other esters are treated with a substantially anhydrous saponifying agent in the presence of an organic liquid diluent, *e.g.*, kerosene, that is chemically inert to the materials present, and is insoluble in and a non-solvent for the resulting alcoholic component, the latter and the diluent being subsequently recovered by distillation at the combined partial pressures of the two. S. S. Woolf.

Production of emulsifying agents. J. H. Disney and J. C. Kernot (E.P. 274,142, 15.1.26).—An emulsifying agent for use with bituminous substances, oils, etc. consists of glue or gelatin (which has been purified by washing, treating repeatedly with alkali, rewashing, and heating repeatedly with dilute acid), mixed with saponifiable material, *e.g.*, resin, tallow, vegetable or animal oil, at a temperature of 40—50°. A suitable resinous material is the liquid resin obtained in the manufacture of sulphite wood pulp, a preferred mixture being 1 pt. of treated glue, 1 pt. of water, and 2 pts. of liquid resin. S. S. Woolf.

Decomposing emulsions in the splitting of fats and oils, and recovering the splitting agent. G. Petroff (E.P. 274,741, 1.2.27).—The emulsions formed in the splitting of fats and oils by various sulpho-acids are broken by the addition of 0·1—0·5% of calcium sulphate. The sulpho-acids may be regenerated from their calcium salts or double salts with calcium sulphate formed in the process, or the salts themselves may be used as splitting agents. S. S. Woolf.

Production of margarine (E.P. 274,949).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Absorption of ultra-violet light by paint vehicles. G. F. A. Stutz (Ind. Eng. Chem., 1927, **19**, 897—901).—The ultra-violet transmission of thin dry films of various oils, varnishes, and lacquers, as well as the change in absorption caused by exposure of the films to sunlight and to the mercury arc, were measured, using a quartz spectrograph. A number of continuous spectrograms of the tungsten spark under water, taken through wet and dry, raw and treated, linseed oil films are also given. Raw linseed oil is quite transparent to ultra-violet light; heat-bodied oils and heavy-bodied air-blown oils are more opaque. Varnishes are quite opaque, but clear nitrocellulose is transparent, opacity being afforded by adding plasticisers and resins. The effect of ultra-violet light on the various films is discussed, with special reference to weathering tests. S. S. Woolf.

Action of cathode rays on drying oils. J. S. Long and C. N. Moore (Ind. Eng. Chem., 1927, **19**, 901—903).—The changes in n_D, iodine value, mol. wt., and hexabromide value when thin oil films were exposed to high-voltage cathode rays for various periods are tabulated for linseed, perilla, and tung oils that had been subjected to preliminary treatment, *e.g.*, heating, blowing, boiling with driers. Mol. wt. increases and iodine value decreases usually on exposure to the rays, whilst n_D increases for linseed and perilla oils but decreases for tung oil, pronounced bleaching occurring in all cases. The improvement in drying time observed is attributed to isomeric change induced by the cathode rays. S. S. Woolf.

Zinc compounds as driers. F. Wilborn (Farben-Ztg., 1927, **32**, 2654—2655).—The statements by Rhodes and others (cf. B., 1923, 1233 A; 1926, 201) that zinc compounds do not increase the rate of absorption of oxygen by linseed oil are not confirmed. The use of zinc driers in conjunction with lead or cobalt driers is recommended, accelerated drying action and improved mechanical properties of the film resulting, but manganese-zinc combinations were not satisfactory. S. S. Woolf.

Mechanical properties of varnish films etc. H. Wolff and G. Zeidler (Farben-Ztg., 1927, **32**, 2708—2710).—The conclusions to be drawn from measurements of tensile strength, elasticity, etc. of detached films are discussed, the importance of changes in these properties with age being stressed. The preparation of films by the application of the varnish etc. to be examined to coloured gelatin foil which is dissolved away with warm water when the coating is dry, is preferred to the other available methods, and details are given. The method of expressing results is described and some typical curves are reproduced, illustrating the loosening of structure undergone by a film on exposure to ultra-violet light, and the irregular extension of a nitrocellulose lacquer. S. S. Woolf.

Degree of saturation of the resin acids. III. B. M. Margosches, K. Fuchs, and W. Ruziczka (Chem. Umschau, 1927, **34**, 215—217; cf. B., 1927, 228, 371).—Mainly a theoretical discussion of the mechanism of the production of halogen acids during the determination, by various methods, of the iodine values of resin acids and allied substances. It is considered that the production of acids in the Hübl and Wijs methods is a secondary reaction resulting in the incipient re-formation of the double linking, but probably a primary reaction in the Margosches–Hinner–Friedmann rapid method (*loc. cit.*). Further evidence, based on the determination of "Plussäure" values, is adduced to demonstrate the analogy between pinene and abietic acid. E. Holmes.

Linseed oil. Eastman and Taylor. **Linseed and perilla oils.** Long and others.—See XII.

Patents.

Bituminous paints and like coating and impregnating compositions. Colas Products, Ltd., and

F. Levy (E.P. 274,955, 28.4.26).—Paints or impregnating compositions containing the reversed bitumen-in-water emulsions or jellies described in E.P. 251,323 (B., 1926, 595) are claimed. Either oil or water paints may be incorporated with the emulsion, reversal of which may take place before or after the incorporation.

S. S. Woolf.

Coating metal and like articles. K. Loos, M. Lehnig, C. Henning, and R. Dassdorf (E.P. 274,333, 15.11.26, and 274,355, 11.1.27).—(a) A smooth, chemically resistant coating is given by a mixture of artificial resin, water-glass, clay or metallic oxides, and silicic acid compounds, the other components being added to the artificial resin at any stage before the final hardening. (b) The final hardening is carried out in the absence of air, *e.g.*, in a bath of oil of high b.p. S. S. Woolf.

Synthetic resins of the phenol-aldehyde type. A. A. Drummond (E.P. 274,581, 26.4.26).—Phenol-aldehyde resins, made by any known method and whether initially in solution or not, are treated with the vapour of an organic solvent of lower b.p. than water, and preferably one with which the resin is miscible, *e.g.*, alcohol, until no further free phenol is removed. The solution of purified resin may be used as such or may be heated *in vacuo* at a temperature below 80° to remove the solvent, which is subsequently recovered.

S. S. Woolf.

Manufacture of moulded phenolic compositions. E. C. R. Marks. From Bakelite Corp. (E.P. 275,011, 12.7.26).—"Cured" moulded phenolic compositions are subjected to further heating for 48—80 hrs. at 125—135°, the dielectric strength of the product at 100° being increased by the treatment to at least 50% of that at 20°. S. S. Woolf.

Manufacture of condensation products of carbamide or its derivatives and formaldehyde. F. Pollak (E.P. 248,729, 17.2.26. Conv., 3.3.25).—Hydrophobic resins are obtained when organic compounds capable of forming resinous condensation products with formaldehyde in an acid medium, *e.g.*, thiocarbamide, phenol, are added to the initial condensation product of carbamide etc. and formaldehyde (*i.e.*, before the "gelatinisation phase" of the first stage commences) and the mixture is further heated in the presence of free hydrogen ions until, on cooling, the resin is precipitated. The mixture may be neutralised or rendered slightly alkaline before cooling since resins produced in this manner retain their hydrophobic qualities in both acid and alkaline solutions. S. S. Woolf.

Manufacture of condensation products from carbamide or a derivative thereof and formaldehyde. Soc. Chem. Ind. in Basle (Ges. für Chem. Ind. in Basel) (E.P. 253,094, 21.5.26. Conv., 5.6.25).—The products are condensed in the presence of active carbon, which is removed at any desired stage of the condensation. An alcohol, ketone, ether, or ester, or mixture of any of these may be present as a retarding agent or diluent, particularly in the conversion of a product at a lower stage of condensation into one at a higher stage in the presence of acid. Effective control of the various stages of the process may be obtained by carrying out the condensation at ordinary temperatures.

S. S. Woolf.

Production of resinous condensation products from amines of the aromatic series. [Resists for batik dyeing.] A. L. Mond. From I. G. Farbenind. A.-G. (E.P. 274,155, 15.3.26).—By condensing an arylamine with formaldehyde in aqueous alcohol in presence of an arylamine salt (up to 10% of the total amine used) the yield of resin is increased and the softening point of the product is raised by an amount depending on the proportion of salt present. Technical formaldehyde contains formic acid, and mineral acid or formic acid may be added to make up the desired quantity. The amount of amine salt necessary is less at b.p. than at lower temperatures. The softening point of the resin is further raised by using 25—50% excess of aldehyde. Volatile matter may be removed in a vacuum. The resins, being stable to alkali, are suitable as resists in batik dyeing, and may be removed from textiles by means of organic solvents, weak acids, or hot water; resins softening below 60° are preferred for this purpose.

C. Hollins.

Manufacture of a cement suitable for linoleum etc. A. B. Craven, C. S. Bedford, and Yorkshire Dyeware & Chem. Co., Ltd. (E.P. 274,300, 30.8.26).—From 20 to 60% of rosin with or without the addition of other suitable resins is incorporated with waste or unused vulcanised rubber and the mixture heated at 110—135°, a definite change taking place in the rubber permitting the formation of a completely homogeneous plastic cement which may be blended with cork, wood, pigments, etc. in the manufacture of linoleum. The rubber may be softened by the use of organic solvents, *e.g.*, xylene, and such other softening agents as "sharp oil," "dead oils," tricresyl phosphate, vegetable oils, etc.

S. S. Woolf.

Manufacture of a cement suitable for linoleum etc. A. B. Craven, C. S. Bedford, and Yorkshire Dyeware & Chemical Co., Ltd. (E.P. 274,722, 3.1.27. Addn. to E.P. 274,300; cf. preceding abstract).—The semi-fluid resinous material obtained from gutta-percha or from balata by extraction with light petroleum etc. or the solid resin obtained from the semi-fluid by oxidation at 100° is incorporated with vulcanised rubber in accordance with the original patent. S. S. Woolf.

Preparation of pigments and paints. J. Blumenfeld, Assr. to Blumann Rare Earths Chemical Co. (U.S.P. 1,639,423, 16.8.27. Appl., 22.4.26. Conv., 22.4.25).—See E.P. 256,302; B., 1926, 889.

Production of coats of lacquers, lacquer and basic oil paints, etc. A. Pahl (E.P. 275,477, 7.1.27).

Colour lakes (E.P. 274,627). **Azo dyes** (E.P. 255,072). **Reduction of nitro-compounds** (E.P. 274,562).—See IV.

Emulsifying agents (E.P. 274,142).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Modifications [in rubber technology] resulting from applications of the antioxygenic theory. J. Dugué (Compt. rend., 1927, **185**, 389—391).—In view of the impracticability of collecting the rubber latex in the absence of air the immediate addition of antioxygens is suggested, to supplement the natural antioxygens present in event of deficiency or partial destruc-

tion. The plasticity and adhesivity of fresh latex, containing 20—25% of moisture, and the loss of plasticity occurring when the product acquires the elasticity of rubber indicate that the polymerides formed in the ageing of rubber are more or less rigid and brittle and not elastic. On this view the cold oxidation or "ageing" of rubber is exactly analogous to vulcanisation, and treatment which effectively prevents autoxidation should also prevent vulcanisation. In vulcanisation, however, a third process of depolymerisation can also take place, with a restoration of elasticity. In vulcanisation under pressure, however, the oxygen present is unable to escape and in the cold catalyses the polymerisation of the rubber. Proposals are made to utilise the plasticity and adhesivity of the freshly-prepared latex for moulding etc. R. Brightman.

Patents.

Producing aldehyde amine condensation products [vulcanisation accelerators]. Grasselli Chem. Co., Assees. of I. Williams and W. B. Burnett (E.P. 263,853, 8.3.26. Conv., 13.3.25).—One mol. of primary or secondary amine reacts with a number of mols. of higher aliphatic aldehyde, water being eliminated, with the formation of highly unsaturated products (viscous liquids or non-crystalline solids), the properties of which change continuously with changing proportions of amine and aldehyde. They are used as vulcanisation accelerators, especially when formed from primary amines and aldehydes in which the α and β carbon atoms are joined by a single linking. As the proportion of aldehyde in the product increases, the "curing" power increases to a maximum and then decreases. The 22 examples given include the preparation of the 2 : 1, 3 : 1, 5 : 1, 7 : 1, 10 : 1, and 15 : 1 heptaldehyde-*n*-butylamine compounds. B. Fullman.

Manufacture of liquefied crude rubber. H. P. Butler (E.P. 274,742, 2.2.27).—Crude rubber is dissolved in benzene etc., carbon tetrachloride is added to render the mixture non-inflammable, and the temperature is raised to 100° for 2—3 hrs. The mixture may be used alone or with the addition of resins, oils, thinners, pyroxylin, etc. as surfacing compositions.
S. S. Woolf.

Process and apparatus for regenerating vulcanised rubber. Soc. Ital. Pirelli (E.P. 260,620, 29.10.26. Conv., 31.10.25).—Combined and uncombined sulphur are removed from ground vulcanised rubber scrap by heating with a solution of caustic soda or other alkali, of medium concentration and in amount slightly in excess of that corresponding to the total sulphur present at ordinary pressures. S. S. Woolf.

Regeneration of vulcanised rubber. C. O. Carlsson (E.P. 274,387, 15.3.27).—Scrap or waste vulcanised rubber is dissolved in tar oil at about 150°, and the dissolved rubber is precipitated by suitable agents, *e.g.*, alcohol, after removal of mineral matter by centrifuging, if desirable. The tar oil and precipitating liquid are subsequently recovered. S. S. Woolf.

Finishing rubber articles. L. Mellersh-Jackson. From L. Candee & Co. (E.P. 274,357, 15.1.27).—The tackiness of the finish produced on rubber articles by coating them with a vulcanisable varnish (usually consisting of a drying oil, sulphur, and a resin) is removed by a superficial treatment with a halogen or a compound comprising a halogen and a member of the sulphur group, excess of halogen etc. being neutralised.
S. S. Woolf.

XV.—LEATHER; GLUE.

Effect of hydrion concentration of tan liquors on absorption of tannin by hide. J. G. Parker and J. A. Gilman (J. Soc. Leather Trades' Chem., 1927, **11**, 213—220).—Pieces of fresh ox hide were limed, unhaired, fleshed, delimed in boric acid solution, washed, and tanned in liquors of p_H 5·0, 4·0, 3·0, 2·5, and 2·0 respectively. The tan liquor was prepared from valonia, myrobalans, mimosa bark, and chestnut extract. The pieces were completely penetrated after 21 days in the liquors of p_H 5·0, 4·0, and 3·0, after 28 days at p_H 2·5, and after 36 days at p_H 2·0. The pieces tanned at p_H 2·0 and 2·5 contained more absorbed matter after 21 days than the other pieces which were completely penetrated so that decreased rate of penetration does not necessarily mean a decreased degree of absorption. The leather tanned at p_H 2·0 and 2·5 was of bad colour and harsh. The leather tanned at p_H 3·0 appeared to have the properties most desirable in sole leather, viz., firmness without brittleness, good substance and colour, and a fairly high tannin content. With a high acidity there is less tendency to bloom deposition. Above p_H 3·0 there was a heavy deposit of bloom on the leather and in the liquors. The greatest yield of leather is obtained at p_H 3·0.
D. Woodroffe.

Action of sulphuric acid on leather. D. Woodroffe and F. H. Hancock (J. Soc. Leather Trades' Chem., 1927, **11**, 225—227).—In tests on pieces of sumach-tanned skiver and East Indian tanned sheepskin treated with solutions of sulphuric acid of different strengths, the acid content of the leather was found to increase and the tensile strength and stretch to diminish with decreased p_H value of the acid solutions. The weakening effect of solutions of $p_H > 2·0$ was appreciable but not serious. The presence of 1% of free sulphuric acid in the East Indian tanned sheep-skin did not cause serious damage, although it did in the skiver.
D. Woodroffe.

Batch B.14 hide powder. D. Burton (J. Soc. Leather Trades' Chem., 1927, **11**, 227—233).—Serious differences have been obtained in tannin analyses using B.14 powder as compared with B.13. Treatment with buffer solution of p_H 5·0 caused it to give fair agreement, but the non-tans of quebracho and blended extracts were lower. By treating it with a buffer solution of p_H 5·6 it gave higher non-tans with these extracts than B.13. Probably p_H 5·6 is too high. D. Woodroffe.

Determination of total sulphur dioxide set free by acid from a bleaching extract. (Report of Committee of Society of Leather Trades' Chemists.) D. Burton (J. Soc. Leather Trades' Chem., 1927, **11**, 221).—The committee recommends the following provisional method for the determination of the total sulphur dioxide set free by acid from a bleaching extract. About 1·2 g. of the bleaching extract are dissolved in 100 c.c. of distilled water, and boiled with about 3 g. of phosphoric

acid in an atmosphere of carbon dioxide so that 50 c.c. distil over into 25 c.c. of 0·1*N*-iodine. The excess iodine is titrated with 0·1*N*-sodium thiosulphate solution. The Burton and Charlton apparatus (cf. B., 1927, 20), or one of which sketch is given, should be used.

D. WOODROFFE.

Titration method for evaluating enzyme bates. J. SCHNEIDER, JUN., and A. ULĈEK (Collegium, 1927, 342—349).—The bate (5 g.) is extracted with 1 litre of water containing 5 g. of ammonium sulphate and 2 g. of ammonium chloride at 18°. 30 c.c. of a standard casein solution and 20 c.c. of water are measured into each of seven flasks placed in a thermostat at 40°, and 50 c.c. of water into each of seven other flasks. Different amounts of bate infusion are now pipetted into the first set of flasks and corresponding amounts into the other set containing water, and serving as blank tests. After 1 hr. the unchanged casein is precipitated with 100 c.c. of a solution of Glauber salt, 45 c.c. of formaldehyde are added to each filtrate, which is then titrated with 0·1*N*-sodium hydroxide until a violet colour appears. The differences in the numbers obtained by titrating the filtrates and blanks, respectively, are plotted against the amount of bate used and compared with the results for a standard bate. D. WOODROFFE.

Digallic acid and tannin as mordants. VIKTOROV.—See VI.

PATENTS.

Treatment of hides and skins preliminary to tanning. R. BOTSON (E.P. 272,195, 4.8.27. Conv., 2.6.26).—Hides and skins are delimed, before tanning, in weak acid baths, such as 1 pt. of acetic acid in 2000 pts. of water, containing a very small proportion of nitrobenzene (0·1% on the acid used).

D. WOODROFFE.

Preparation of chamois leather. A. J. CLERMONTEL (F.P. 595,954, 31.3.25).—Raw goat- and sheep-skins, after liming for 12 hrs. with lime alone, are unhaired, fleshed, coated on the flesh side with a 15% aqueous solution of sodium carbonate, and again after 5 hrs., and piled for 6 hrs. The skins are then rinsed in water, drained for 20 min., partially dried by drumming in sawdust or gypsum, oiled several times with linseed oil, and exposed to sunlight in the open or a warm room at 40°. 24 hrs. are necessary for the tannage. Finally, the skins are freed from surplus oil by drumming in sawdust or gypsum-meal for 3 hrs. D. WOODROFFE.

Manufacture of tanning agents. I. G. FARBENIND. A.-G. (G.P. [A] 438,199, 24.7.24, [B] 438,200, 7.9.24).—(A) Dry powdered wood charcoal containing 9% or more oxygen is mixed with water and treated with nitric acid or an oxide of nitrogen at 70—90° until entirely dissolved, requiring several days. The excess nitric acid or nitrous gases is distilled off by heating for about 30 hrs., avoiding excessive heat. The product is soluble in water and can be used with or without previous neutralisation for tanning. (B) The strongly acid properties of the products obtained by (A) are repressed by mixing them with neutralising agents, *e.g.*, sodium carbonate solution, other synthetic or natural tanning materials. D. WOODROFFE.

Tanning animal hides with metallic salts. E. CHICOINEAU (F.P. 587,202—3, 19.12.23).—The pelts are treated in neutral, acid, or alkaline solutions with salts of antimony or zinc (*e.g.*, 4—6% of zinc chloride and 5—10% of salt). After penetration, they are horsed up for 1—2 days, treated with water or weak solutions of hydrogen peroxide, and then immersed in a very dilute solution of sodium carbonate or other alkali salt, which should be gradually strengthened until the metallic salts in the pelts are precipitated on the hide fibres. D. WOODROFFE.

Process of chrome tanning. A. SAUTOT (F.P. 579,207, 2.6.23).—Liquid sodium bisulphite is added to a mixture of sulphuric acid and potassium dichromate solution until the liquor is distinctly green. The product, when cold, is used for tanning pelts.

D. WOODROFFE.

Treatment of horn etc. F. HOMBERG, Assr. to AMER. NUPLAX CORP. (U.S.P. 1,636,818, 26.7.27. Appl., 30.12.24).—Horn is soaked with hot (but not boiling) acid, then kept, and the treatment repeated until the horn is softened, when it is washed with water, dried, and comminuted. D. WOODROFFE.

Stuffing of chrome-tanned heavy leather. R. H. PICKARD, D. J. LLOYD, and A. E. CAUNCE (U.S.P. 1,638,877, 16.8.27. Appl., 22.8.25. Conv., 27.8.24).—See E.P. 243,438; B., 1926, 138.

Obtaining solidified granules or pearls [of glue etc.] from liquids. W. WACHTEL, Assr. to A.-G. FÜR CHEM. PROD. VORM. H. SCHEIDEMANDEL (U.S.P. 1,638,669, 9.8.27. Appl., 29.6.25. Conv., 15.12.24).—See E.P. 244,406; B., 1926, 990.

XVI.—AGRICULTURE.

Soil phosphorus studies. I. Colorimetric determination of organic and inorganic phosphorus in soil extracts and the soil solution. F. W. PARKER and J. F. FUDGE. **II. Concentration of organic and inorganic phosphorus in the soil solution and soil extracts, and availability of organic phosphorus to plants.** W. H. PIERRE and J. W. PARKER. **III. Plant growth and absorption of phosphorus from culture solutions of different phosphate concentrations.** F. W. PARKER (Soil Sci., 1927, **24**, 109—117, 119—128, 129—146).—I.—For the determination of phosphorus in small quantities the method of Denigès (B., 1920, 781 A) is more sensitive than that of Fiske and Subarrow (A., 1926, 443). The presence of considerable amounts of silica does not affect the accuracy of the method. The technique for determining total and inorganic phosphorus is described. II.—In displaced soil solutions the concentration of organic and inorganic phosphorus averaged 0·47 and 0·09 pts. (PO_4) per million, respectively. Plants absorbed all the inorganic phosphate, but none of the organic phosphate from soil solutions and extracts. The bearing of this unavailability of organic phosphate in the interpretation of plant nutrition experiments is discussed. III.—In culture solutions maximum growth of maize and soya beans was obtained with a concentration of 0·50 pt. (PO_4) per million. It is estimated that one half of this

concentration would suffice if this could be maintained continuously during plant growth. The solid phase of the soil would appear to play an important part in the phosphorus nutrition of plants, but the mechanism whereby a concentration of inorganic phosphate so much greater than that of displaced soil solutions is maintained cannot be explained by the familiar theories of soil chemistry. A. G. Pollard.

Modification in the solubility of phosphoric acid and in the biological properties of the soil observed during the fallowing of soil previously dried in the air. [I.] A. Lebediantzef (Compt. rend., 1927, **185**, 397—399; cf. B., 1927, 709). [II.] A. T. Schlœsing (*ibid.*, 399—400).—I.—Determinations of the phosphoric acid soluble in water, in 2% acetic acid, and in 0·5% oxalic acid on dried and undried samples of different soils (fallow, at 20 cm. depth, triennial rotation at 20 cm., and at 40 cm. depth) show that the desiccation produces marked changes in the phosphoric acid distribution when the samples are left to fallow at the ordinary temperature and with a humidity about 30%. The water-soluble phosphoric acid is increased at first by the drying, but in 25 days decreases to approximately the content of the undried sample, and does not further increase. The phosphoric acid soluble in acetic acid and in oxalic acid shows an increase on the dried sample persisting over a much larger part of the fallow period. The bacteria content of the soil decreases on drying, and remains low during the 2—3 months' fallow period. On the other hand, the production of carbon dioxide is increased by desiccation. II.—The temporary nature of the increase in soluble phosphoric acid content on drying is attributed to (1) the difference in the soils used by Lebediantzef and by Schlœsing and Leroux (B., 1927, 343), (2) different methods of washing employed in extracting the phosphoric acid, and (3) the high humidity (30%) in which Lebediantzef's samples were kept after drying. It is concluded that soils kept slightly moist (humidity 6—7%) after desiccation manifest an increased soluble phosphoric acid content effective for crops during a period of three months after desiccation.

R. Brightman.

Action of superphosphate and Rhenania phosphate on soil. L. von Kreybig (Z. Pflanz. Düng., 1927, **A 9**, 224—235).—The author has investigated layer by layer the effect of superphosphate and "Rhenania" phosphate on the chemical and biological properties of an acid soil. Both fertilisers were applied to the surface at rates usual in practice, and had little effect on the acidity relationships, except that superphosphate increased slightly the hydrolytic acidity of the layer 0—1 cm. The biological activity as evidenced by nitrogen fixation was increased more by the basic Rhenania phosphate than by the superphosphate. The effect of both dressings is confined to the surface layer, and it is shown that the 1% citric acid-soluble phosphate in this layer is greater with Rhenania phosphate than with superphosphate. The author emphasises the localised character of the action of both dressings, and the importance of mode of application and mixing with the soil. G. W. Robinson.

Determination of humic acid [in soils]. O. Schaill (Landw. Versuchs-Stat., 1927, **105**, 209—230).—In a comparison of existing methods for determining humic acid, closest agreement was found between the methods of Tacke and Suchting and of Albert and the titration value of the active acidity. Colorimetric methods depending on the preparation of aqueous soil extracts give results depending on the p_H value of the extracts. The "loss on ignition" method of determining total organic matter in soils yields results closely parallel to those obtained by carbon dioxide measurements. Comber's method is of value in highly organic soils only. The preliminary drying of the soil sample in the Tacke method involves possible alteration in the nature of the organic matter present. A. G. Pollard.

Determination of the p_H values of soils. K. Nehring (Landw. Versuchs-Stat., 1927, **105**, 231—240).—In preparing soil extracts for p_H determinations the nature of the filter paper frequently affects the results obtained. Only the finest paper should be used, and pre-washing with distilled water is essential. Soil extracts prepared with potassium chloride solution are less liable to be affected in this way. Quinhydrone for electrode work must be highly purified, as many commercial samples have an acid reaction. A. G. Pollard.

Effect of manuring a crop on the vegetative and reproductive capacity of the seed. B. V. Nath and M. Suryanarayana (Mem. Dept. Agric. India, 1927, **9**, 85—124).—In experiments with "ragi" (*Eleusine coracana*), "cholam" (*Andropogon Sorghum*), "pani" (*Panicum miliaceum*), and wheat, the seed obtained from manured plots gave higher yields than seed from unmanured plots. In general, seed from plots which had received cattle manure gave better results than when complete minerals were used. The "cattle manure" seed was also found to be superior in animal nutritive value to "mineral manure" and "no manure" seed. This superiority is held to be connected with vitamin content. Experiments with farmyard manure and extracts of it indicate that organic manures, in addition to improving soil texture and supplying plant food, also supply "auximones," or substances which behave towards plants in the same manner as vitamins. Marked increases were obtained with small applications of yeast. It is held probable that the microbial population of the soil contributes to the plant a stimulant which is ultimately passed on to the animal.

G. W. Robinson.

Influence of manures and organic residues on plant growth. G. A. Newton and K. B. Daniloff (Soil Sci., 1927, **24**, 95—101).—The reduced plant growth resulting from the addition to soil of organic material of low nitrogen content may be obviated by the use of sodium nitrate. A. G. Pollard.

Absorption of nutrients by plants from finely-ground basalt. J. Sachse (Z. Pflanz. Düng., 1927, **A 9**, 193—223).—In water and sand cultures with peas, barley, oats, and rye, it was found that whilst the potassium of finely-ground basalt was relatively available, the calcium and phosphorus were only slightly available. The effect of treatment of the ground basalt with hot water, 1% sulphuric acid, and 1% ammonia,

respectively, was also investigated in experiments with peas. The availability of the magnesium, potassium, and phosphorus was increased to the greatest degree by the ammonia treatment, and that of the calcium and silica by hot water treatment. The yield and the absorption of nutrients were increased by addition of calcium carbonate. The results of extraction of the ground basalt with a number of different reagents are given. G. W. ROBINSON.

Antagonism between chlorides used in large amounts and sulphates in the development of wheat and oats on a large scale. E. BLANCHARD and J. CHAUSSIN (Compt. rend., 1927, **185**, 218—220).—Excessive use of fertilisers rich in chlorides is to be avoided, as these cause an almost complete disappearance of sulphur compounds in the plants, and are a danger to the crop in dry seasons. B. W. ANDERSON.

Experiments with the Neubauer seedling method. T. STECHE (Z. Pflanz. Düng., 1927, **A 9**, 236—249).—The Neubauer seedling method gives satisfactory results if the amounts of potassium and phosphorus taken up are compared, not with the amounts originally present in the seeds, but with the amounts found in a blank experiment, because in the latter case there is an actual loss of potassium and phosphorus to the soil. In the case of acid soils, it is better to determine the nutrient requirements by the Neubauer method after the acidity has been corrected by liming. G. W. ROBINSON.

Production of nodules on different parts of the root systems of lucerne plants growing in soils of different reaction. P. E. KARRAKER (Soil Sci., 1927, **24**, 103—107).—The poor development of nodules on lucerne roots grown in acid soil is the result of the direct action of the acid soil on the organisms within the nodule. Acid soils do not appear to influence the plant or the free living organisms in the soil in a manner to affect the course of nodule production.

A. G. POLLARD.

Selection for quality of oil in soya beans. L. J. COLE, E. W. LINDSTROM, and C. M. WOODWORTH (J. Agric. Res., 1927, **35**, 75—95).—Selective breeding from a commercial variety of soya bean resulted in the isolation of two definite strains—a tall, late type, bearing oil with iodine value 133·7, and a dwarf early type, with oil of iodine value 124·9. High iodine values in soya bean oil appears intimately connected with late maturity in the plant. No correlation exists between the quality of oil from soya plants and the yield per plant. The quantity of oil produced cannot be markedly increased by selective breeding. A. G. POLLARD.

Lubricating oils as insecticides in dormant spraying. E. L. GREEN (Ind. Eng. Chem., 1927, **19**, 931—935).—A series of lubricating oils emulsified by a solution of potash fish-oil soap in crude cresylic acid was tested for insecticidal power by using as sprays in orchards infested with San José scale and orchard leaf roller. The toxicity bears no relation to the viscosity of the oils, but is greatest in the portion that distils between 240° and 300°/40 mm. Owing to the low field temperatures and the high boiling ranges of the effective oils, it is improbable that the oils attack the tissue of the insects as vapour. Oils with an asphalt or a paraffin base form equally good insecticides, whilst those which have been decolorised with sulphuric acid are likely to be more effective than the untreated oils. F. R. ENNOS.

Increasing life activities of plants. DENNY.—See XIX.

XVII.—SUGARS; STARCHES; GUMS.

Glucose and its industrial preparation by acid and by enzymic saccharification of starch. G. MEZZADROLI and A. NARDELLA (Zymologica, 1927, **2**, 49—62, 73—89).—A summary is given of the preparation and properties of commercial glucose, and experimental results are described which show that, by the enzymic saccharification of starch by means of *Mucor*, a greater proportion of the raw material is utilised than when the conversion is effected by acid. In the latter case, appreciable proportions of the starch and of the glucose formed are transformed into humic substances and non-saccharine products, and the unattacked grain residue is almost useless for cattle food. On the other hand, the juices obtained by enzymic saccharification of grain contain only traces of caramelised products, and the residues serve as a valuable feeding material.

T. H. POPE.

Viscosimetric determinations with low-grade sugar refining products. F. DÍAZ AGUIRRECHE (Anal. Fís. Quím., 1927, **25**, 178—181).—The author proposes to determine the viscosity of concentrated sugar solutions by measuring the time taken for a float of known density to rise through a given distance in a column of the solution, corrections being applied for temperature.

G. W. ROBINSON.

XVIII.—FERMENTATION INDUSTRIES.

Relationship of ready-formed soluble carbohydrates in malt to extract. H. E. DRYDEN (J. Inst. Brew., 1927, **33**, 420—424).—It is suggested that a better idea of the composition of the extract is obtained and that the relationship of all malts can be better compared if the ready-formed soluble sugars, which are arrived at by deducting 4% from the total matters soluble in cold water, are expressed in lb. per 100 lb. of extract and also as a percentage in the analytical report. On this basis foreign malts show a lower proportion of ready-formed soluble sugars, and consequently the composition of the wort from foreign malts is more under control. C. RANKEN.

PATENTS.

Fermentation and maturation of beer. HANSENA A.-G., and L. NATHAN (E.P. 274,225, 26.4.26).—The Nathan process for the production of beer is improved by carrying out the primary fermentation below 6°, with about 2 litres of yeast per hectolitre, until deposition of the yeast occurs, the attenuation being carried to within $1\frac{1}{2}$—1% of residual fermentable matter. To prevent any further activity on the part of the deposited yeast, the settling space of the fermentation vessel is strongly cooled from the commencement of yeast deposition, preferably to 1°. The maturing or secondary fermentation is effected at 10—25° in the presence of a relatively small quantity of well-aerated, non-germinating

yeast, which may be either the remainder of the yeast produced in the primary fermentation and subsequently aerated, or else taken as fresh yeast from the yeast tank. C. Ranken.

Pasteurisation of beer. Hansena A.-G., and L. Nathan (E.P. 275,090, 26.4.26).—The finished beer, after expulsion of carbon dioxide by heating at a relatively low temperature, is heated to the pasteurising temperature, subsequently cooled to approximately —1°, and carbonated. It is then bottled and pasteurised again in the bottles at a temperature about 1° lower than that at which the first pasteurisation took place.

C. Ranken.

Fermentation of cellulose. H. Langwell (U.S.P. 1,639,571, 16.8.27. Appl., 7.1.21. Conv., 8.1.20).—See E.P. 161,294 ; B., 1921, 405 a.

XIX.—FOODS.

Wheat and flour studies. XI. Extraction of proteins from wheat flour. P. F. Sharp and B. L. Herrington (Cereal Chem., 1927, 4, 249—260).—The separation of wheat proteins into four fractions by the official American method is not exact owing to overlapping solubilities. As a basis for formulating a more accurate procedure, the influence of time, concentrations, temperatures, etc. in the successive extraction method has been examined. For the extraction of albumin, globulin, etc., 6 g. of flour and 100 c.c. of 5% potassium sulphate solution were shaken for 1 hr., and centrifuged, and, after filtering, nitrogen was determined in 50 c.c. of the filtrate by the Kjeldahl method. Gliadin was determined in the residue by shaking for 1 hr. with (150—X) c.c. of 70% alcohol (where X is the weight of potassium sulphate solution retained in the residue after filtration), and, after centrifuging, nitrogen is determined in 50 c.c. of the supernatant liquid. Protein-nitrogen in the alcoholic residue was determined by the Kjeldahl method, using 10 c.c. of sulphuric acid, 0·7 g. of mercury, and 10 g. of potassium sulphate, followed by 35 c.c. of sulphuric acid when the frothing had subsided. In calculating the glutenin, allowance was made for the small amounts of protein carried forward by the potassium sulphate solution and alcohol retained in the residues. The protein factor is 5·7 for each fraction. By this method, the alcohol-soluble protein fraction is much lower and the glutenin percentage much higher than is usually recorded. Extracting 4 g. of flour with alcohol of various concentrations showed that the maximum quantity of protein (7·43%) is removed by 50 vol.-% alcohol when shaking is employed, and when refluxing is performed the amount extracted is increased for all concentrations. Varying the ratio of the weight of flour to the volume of alcohol had no appreciable effect. The amounts of protein removed by 5% and 10% solutions of various salts, followed by shaking and by refluxing with 70% alcohol, were also studied. The maximum amount was extracted by magnesium chloride, and extraction by this salt was more complete with the 10% solution ; in all other cases investigated, the 5% solutions were the more efficient. H. J. Dowden.

Relation of hydrogen-ion concentration of dough to baking properties. E. Grewe and C. H. Bailey (Cereal Chem., 1927, 4, 261—270).—The influence of hydrogen-ion concentration upon the diastatic activity of 17 flours has been investigated. The addition of sufficient lactic acid to reduce the p_H from 5·9 to 5·0 substantially increased the activity in every case, the rate of increase being greatest with flours of high initial activity. The addition of alkali to flours diminishes diastatic activity, and it was observed that increasing the p_H value from 5·9 to 8·5 by adding 3·6 c.c. of 0·1N-caustic soda solution to 10 g. of flour reduced the diastatic activity from 206 to 12 mg. of maltose per 10 g. of flour. The diastatic activity of flours of low initial activity is substantially increased by adding flour milled from sprouted wheat, an addition of 5% being capable of raising the activity of a flour from 125 to 200 units, which is the desirable activity for a flour to be of good baking quality. The loaf volume and crumb texture are also improved by the addition of sprouted wheat flour. The addition of alkali and sufficient sprouted wheat flour to maintain a high diastatic activity demonstrated that the best bread as regards loaf volume and crumb texture is given by doughs in which the p_H has been decreased. These facts indicate that the increase of p_H during dough fermentation results in improved bread because of the accelerated activity of the enzymes. H. J. Dowden.

Factors affecting interpretation of experimental baking tests. M. J. Blish and R. M. Sandstedt (Cereal Chem., 1927, 4, 291—299).— The fermentation tolerance of doughs has been investigated by performing the Werner baking test on doughs fermented from ½—6 hrs. Fermentation tolerance can be accurately judged at one bake, from the crust colour, rise, texture of the bread, etc., provided other influencing factors are maintained constant. Great fluctuations in stability were observed, experimentally milled flours showing " exhaustion " much more quickly than commercially milled flours. To make the results comparable it is recommended that with experimentally milled flours, the sugar content should be increased from 2½% to 5%. Crust colour also gives a valuable indication of the diastatic value. These facts suggest that fermentation tolerance is dependent on the yeast, diastase, or sugar content rather than on the gluten quality or quantity. Oxidising agents act powerfully on gluten, and are largely responsible for the variations in bread-making qualities of flours. A differential baking test, with and without added oxidising agent, indicates readily to what extent the flour can be improved by the addition of these agents. Overtreatment gives symptoms of " age." H. J. Dowden.

Value of experimental [flour] milling test. R. S. Herman (Cereal Chem., 1927, 4, 270—274).—Experimental milling gives results which are strictly comparable with commercial milling, and is of value in ascertaining the nature of wheat, yield, and flour quality, as regards colour and analytical data. The flour can be used for determining baking characteristics and thus provides guidance in blending. H. J. Dowden.

Washing of gluten from flour. D. W. Kent-Jones and C. W. Herd (Analyst, 1927, 52, 439—443).—A number of chemists determined the gluten content of the same flours, using their own tap water and the special solutions of Dill and Alsberg, and under as nearly as

possible identical conditions. It was found that differences caused by using tap water and the special solutions were very small, but large differences occurred between the results of the various workers. Each, however, obtained essentially consistent results, so that the ratio between the nitrogen of the flour and the dried gluten was approximately consistent for each. D. G. HEWER.

Numerical expression for the colour of flour. D. W. KENT-JONES and C. W. HERD (Analyst, 1927, **52**, 443—452).—Definite numerical expressions for the colour factors of flour may be obtained by the successive use of two solvents, viz., a standard grade petrol for extraction of the yellow carotin, and alkaline methyl alcohol for extraction of the reddish-brown pigment, which latter presumably comes from finely powdered offal. The determinations are made against standard solutions in a special colorimeter, and the petrol figure indicates the yellowness, not differentiating high- and low-grade flour, whilst the methyl alcohol figure is the grade figure showing the brightness of the flour and its freedom from minute branny specks. In practice wood spirit may be used for methyl alcohol if the necessary correction for each batch is applied. D. G. HEWER.

Chemical modifications produced in wheat flour by benzoyl peroxide. S. BAGLIONI and L. SETTIMJ (Annali Chim. Appl., 1927, **17**, 351—356; cf. Javillier, B., 1926, 211).—When benzoyl peroxide acts upon wheat flour it is completely decomposed to give oxygen and benzoic acid, which is lost almost completely at 150°. Its bleaching action consists in conversion of carotin into a leuco-compound; at the same time some non-glutinous proteins are converted into proteoses, a probable cause of the rapid maturing of the flour. Carbohydrates, fats, lecithin, phytosterol, etc. remain unchanged, as do the calorific value and the physical constants. E. W. WIGNALL.

Cacao butter substitutes and their detection. A. W. KNAPP, J. E. MOSS, and A. MELLEY (Analyst, 1927, **52**, 452—456).—A criticial review of the methods in use for the detection of illipé butters, true and commercial, and shea butter shows that no one method is satisfactory, but by a comparison of results obtained from the titre of the fatty acids, solubility tests, Tate and Pooley's method, and Bywaters' process (B., 1927, 614), together with a determination of the permanence of any green colour, a fair idea of the presence and proportion of cacao butter substitutes may usually be arrived at. The green colour test depends on the comparative resistance of the green colour of Borneo tallow to bleaching; 30 g. of fat are exposed in a Petri dish 15 cm. diam. for 6 hrs. 6·5 in. below a quartz mercury lamp of uroxameter value 28·0, the time of exposure varying with the strength of the lamp. The fat is then filtered into a test tube and the colour compared with that of a set of standard exposed fats. The method was found most useful for mixtures containing less than 20% of Borneo tallow.

D. G. HEWER.

Effect of common salt on lime water used for egg preserving. J. MILLER (Analyst, 1927, **52**, 457—458).—Common salt added to lime water increases the progressive diminution of strength of the lime water in proportion to the amount added. Even with excess of lime all the lime is completely carbonated in 6 months in the presence of 100 lb. or more of common salt per 1000 gals. It is suggested that the instructions in Special Report No. 26 of the Department of Scientific and Industrial Research, to use 4 pts. of slaked lime to 20 of cold water might be modified, as the solubility of lime is about 1 in 750, and if tap water is mixed with 4 times the amount of burnt lime necessary to saturate, stirred for 5 min., and allowed to settle, a practically saturated lime solution is obtained without further keeping. The addition of 1 pt. of salt, as instructed, equivalent to 500 lb. per 1000 gals., is not only unnecessary but detrimental.

D. G. HEWER.

Ammonia content of cold-storage eggs. H. C. LYTHGOE (Ind. Eng. Chem., 1927, **19**, 922—924).—The average monthly content in ammonia of eggs put into cold storage before July 1 showed a gradual increase from 2·4 mg./100 g. in September to 3·2 mg./100 g. in February. Of 1100 cold-storage eggs collected during the entire season for a period of five years, 95% contained between 2·1 and 4·3 mg. NH_3 per 100 g., the former figure probably representing the minimum content at which it is possible to market eggs preserved in this manner. F. R. ENNOS.

Natural occurrence of boron compounds in cacao and cacao products. S. DODD (Analyst, 1927, **52**, 459—466).—Boron compounds occur naturally in cacao and cacao products and are present in chocolate to the extent of about 0·01% (as boric acid) and in commercial samples of cacao beans and cocoa from 0·0217 to 0·0837%, and in coffee beans to about 0·01%. Boric acid may be satisfactorily determined by Thomson's method, but the best indicator is Sofnol indicator No. 1 with p_H 6·5. For qualitative work turmeric paper is used, and it is advisable in the case of oily substances to remove the bulk of the oil before testing.

D. G. HEWER.

Effect of small amounts of chemicals in increasing the life activities of plants [and ripening of fruit]. F. E. DENNY (Proc. Nat. Acad. Sci., 1927, **13**, 555—561).—When ethylene is added to the air in which oranges and lemons are stored, a concentration of gas not exceeding 1/5000 doubles the rate of production of carbon dioxide, and any green colour in the peel is rapidly converted into yellow. Too high concentrations of ethylene retarded the change of colour. The evidence shows that the ethylene acts in a physiological and not in a directly chemical manner, raising the general level of life activity in the tissues. The stimulative effects also apply to tomatoes, potatoes, apples, etc. Freshly-harvested potatoes if replanted under conditions favourable to growth do not sprout at once, but require a period of after-ripening. Various chemicals induce early sprouting, ethylene chlorohydrin and alkali thiocyanates being specially effective. The substances causing a stimulation of life activity do not appear to be chemically related, they are not used as food, and the mechanism of their action is not understood. R. A. MORTON.

Interpretation of bromatological analyses. A. BEYTHEN, C. HARTWICH, and M. KLIMMER (Anal. Oficina

Quím. Prov. Buenos Aires, 1927, **1**, 91—100).—The authors give standards for the interpretation of analyses of meat and meat products. G. W. Robinson.

Direct determination of sodium chloride in food-stuffs. F. Mach and W. Lepper (Landw. Versuchs-Stat., 1927, **105**, 333—334).—The sample (5·0 g.) is extracted for 30 min. with about 400 c.c. of water and the extract cleared by precipitation with 40 c.c. of a solution containing 50 g. of phosphotungstic acid, 500 c.c. of nitric acid (*d* 1·4), and water to make 1 litre. In the filtered solution chlorine may be determined by the Volhard method with the addition of ether.

A. G. Pollard.

Patents.

Treatment of milk powder [for packing]. W. G. Hawley, Assr. to Merrell-Soule Co. (U.S.P. 1,637,311, 26.7.27. Appl., 29.1.27).—The milk powder, enclosed in a container which is air-tight except for a minute opening, is subjected to a high vacuum and then charged with a mixture of carbon dioxide and nitrogen, so that the uncombined oxygen content of the powder and space within the container is less than 5 c.c. per lb. of powder. F. R. Ennos.

Production of margarine and like edible fats. B. Jirotka (E.P. 274,949, 26.4.26).—The margarine mass, after preliminary disintegration, is fed in opposite streams over cooled rollers between which it is kneaded (pressing and spreading action being produced by the different peripheral speeds of the rollers), the fresh films so formed being removed by scrapers.

S. S. Woolf.

Refining bee honey. W. W. Somerford (U.S.P. 1,636,719, 26.7.27. Appl., 24.8.22).—The natural honey, mixed with a filtering substance and water, is run through heated filter bags and the filtered mass stirred in a vacuum tank until it attains a predetermined consistency.

F. R. Ennos.

Making concentrated fruit juice. W. M. Zorn (U.S.P. 1,636,890, 26.7.27. Appl., 25.7.24).—The juice extracted from the fruit by pressure is filtered to remove pulpy matter and concentrated by freezing to a snow and centrifuging. The concentrated juice is heated to below its b.p., filtered to remove pectin and other undesirable matter, cooled to just above its f.p., and refiltered. F. R. Ennos.

Curing meats. J. Alsberg, Assr. to B. Heller & Co. (U.S.P. 1,635,301, 12.7.27. Appl., 12.6.25).—After being charged with a curing liquid containing sodium chloride, a nitrate, and a nitrite, the meat is completely enveloped in a dry curing medium having a predetermined amount of nitrite for 40—60 days. F. R. Ennos.

Preservation of food-stuffs etc. C. T. J. Vautin and W. G. Whiffen (E.P. 274,164, 6.4.26).—Putrefaction is prevented by protecting the exposed surface of the substance from the atmosphere by means of a porous material impregnated with calcium iodate. In certain cases the iodate may be added direct in the form of a powder, or the substance may be immersed in an aqueous solution of the iodate. C. Ranken.

Treatment of cereal substances. D. W. Kent-Jones and C. W. Chitty, Assrs. to Woodlands, Ltd. (U.S.P. 1,636,569, 19.7.27. Appl., 5.11.25. Conv., 9.12.23).—See E.P. 228,830; B., 1925, 296.

Egg-preservation process and product thereof. A. J. Bellamy, Assr. to Egg Patents Ltd. (U.S.P. 1,639,549, 16.8.27. Appl., 2.9.25. Conv., 13.5.25).—See E.P. 261,194; B., 1927, 92.

Improving the qualities of coffee. O. Gewalt (U.S.P. 1,636,300, 19.7.27. Appl., 16.7.26).—See E.P. 265,865; B., 1927, 315.

Machinery for making aqueous emulsions of dried milk or milk powder. A. C. Bamford (E.P. 275,375, 12.6.26).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Preparation and properties of diethylene glycol dinitrate. W. H. Rinkenbach (Ind. Eng. Chem., 1927, **19**, 925—927).—Diethylene glycol dinitrate, $O[C_2H_4 \cdot NO_3]_2$, is prepared by nitration of pure diethylene glycol with mixtures of nitric and sulphuric acids, the yield, emulsification, and safety of the operation depending on the type of nitrating mixture used. The best yields are obtained by adding the glycol to just sufficient nitrating acid containing 25% of nitric acid and 5—10% of water, keeping the temperature below 10°, pouring the resulting emulsion into three times its weight of ice and water, purifying the ester by washing with ice water and potassium carbonate, and drying over sulphuric acid. Pure diethylene glycol dinitrate is a clear, colourless, odourless liquid of greater fluidity than nitroglycerin; at ordinary temperatures it is completely miscible with nitroglycerin, ethylene glycol dinitrate, ether, acetone, methyl alcohol, chloroform, benzene, toluene, aniline, nitrobenzene, glacial acetic acid, and glycol diacetate. It is immiscible with or only slightly soluble in ethyl alcohol, carbon tetrachloride, and carbon disulphide. Its solubility in water is 4·1 g./litre at 24°, but it is only slightly hygroscopic. At ordinary temperatures it is not saponified by 3% alcoholic potash. It has d_{15}^{15} 1·3908, f.p. —11·3°, n_D^{15} 1·4536, heat of combustion at constant volume 2798 g.-cal./g., at constant pressure 2792 g.-cal./g., vapour pressure at 22·4° 0·007 mm. Diethylene glycol dinitrate can be ignited only by heating above the temperature of the air. It is less sensitive to shock than nitroglycerin or glycol dinitrate, but when mixed with these it can be completely detonated and shows an explosive effect but little less than either of these compounds. The physiological action of diethylene glycol dinitrate on the human system is much less marked than that of nitroglycerin.

F. R. Ennos.

Determination of cincophen (2-phenylcinchoninic acid) and choice of indicators for its titration. S. Palkin (J. Amer. Pharm. Assoc., 1927, **16**, 632—635).—The material may be extracted from a slightly acid aqueous solution by means of a mixture of chloroform and ether, or it may be treated in acid solution with a mixture of potassium bromide and bromate, and the additive compound extracted with ether. The substance is weighed as the hydrobromide, after removal of the excess bromine by repeated evaporation with acetone.

Electrometric titration indicates a p_H range of 6·5—8·5 for the neutral salts, so that cresol-red, phenol-red, or bromothymol-blue are more suitable indicators for the titration with acids than phenolphthalein.
S. I. LEVY.

Critical study of methods of analysis of antipyrine and pyramidone. A. BORLOZ (Helv. Chim. Acta, 1927, **10**, 543—548).—Antipyrine may be accurately determined by the iodometric methods of Bougault (A., 1917, ii, 344) and Kolthoff (A., 1923, ii, 270), other methods being unreliable. For pyramidone none of the five methods proposed is good, the best being titration with sulphuric acid in presence of methyl orange (Pegurier, A., 1905, ii, 871), which gives low results and requires special technique. For mixtures of pyramidone with small amounts of antipyrine, Patein's formaldehyde method (A., 1905, ii, 658) is available for the latter, pyramidone being extracted with chloroform and determined by titration. In a mixture containing much antipyrine, pyramidone is first titrated with acid, excess of picric acid is then added to a second sample and the excess titrated (cf. Pegurier, *loc. cit.*).
C. HOLLINS.

Determination of iodine in extract of thyroid gland. J. F. REITH (Pharm. Weekblad, 1927, **64**, 777—792).—Experiments carried out to trace the causes of loss of iodine during the burning off show that no loss of iodine occurs when a mixture of potassium iodide with a large excess of fusion mixture is heated, unless a very high temperature is attained. Iodine vapour once formed, however, is not completely absorbed by heated fusion mixture unless the temperature is not less than 400°. Hunter's method (A., 1910, ii, 650) is rapid and accurate, but gives results considerably higher than those obtained by using the method of the Dutch Pharmacopœia; in the latter, iodine is lost in the combustion. The sources of error in the Dutch method are pointed out in detail.
S. I. LEVY.

Factors affecting certain properties of a [tobacco] mosaic virus. H. H. McKINNEY (J. Agric. Res., 1927, **35**, 1—12).

Quantitative and purification methods in [tobacco] virus studies. H. H. McKINNEY (J. Agric. Res., 1927, **35**, 13—38).

Bismuth salts. CHEMNITIUS.—See VII.

Hydnocarpic acid. PERKINS and others.—See XII.

PATENTS.

Production of oxygen-containing aliphatic compounds [keten, acetaldehyde, etc.]. H. DREYFUS (E.P. 273,622 and 273,810, 26.6.25).—(A) Keten is obtained by passing acetaldehyde vapour over a heated hydrogen-splitting catalyst, *e.g.*, nickel, copper, zinc, etc., at not above 400—500° (preferably between 150° and 300°) and preferably at normal pressure; in the absence of a catalyst the reaction is effected at higher temperatures and at 5—50 atm. pressure. The keten may be collected as such, or converted into acetic acid or/and anhydride by treatment with water or/and acetic acid. Acetic acid or/and anhydride are obtained directly by mixing the acetaldehyde with water or/and acetic acid prior to treating it as above. The acetaldehyde may be of any origin or produced by reducing the vapour of acetic acid (glacial, concentrated, or dilute) according to E.P. 273,810. (B) Dilute or waste acetic acid is reduced to acetaldehyde by passing its vapour, together with hydrogen or gases containing it, with or without carbon monoxide, *e.g.*, water, town or producer gas, etc., over reduction catalysts such as nickel, iron, tin, platinum, palladium, etc. or/and contact substances such as pumice, glass-wool, sodium chloride, etc., singly or mixed. The hydrogen is preferably used in excess of theoretical proportions. *E.g.*, 2 vols. of 15—30% acetic acid are mixed with 1 vol. of hydrogen and passed over finely-divided nickel maintained at 250—300°. The aldehyde is separated by fractional condensation, and may be converted into glacial or concentrated acetic acid by oxidation.
B. FULLMAN.

Manufacturing borneol and *iso*borneol. G. AUSTERWEIL (E.P. 258,901, 27.9.26. Conv., 25.9.25).—Nopinene (1 mol.) reacts in neutral solvents (toluene, xylene, pinene, etc.) with 2 mols. of an organic acid (benzoic or dichlorobenzoic acid etc.), yielding only bornyl esters and pinene, without monocyclic terpenes. The reaction is carried out at an acid concentration not exceeding 30% (preferably 20—25%) at 140—150°, heating for 40—50 hrs., according to the acid concentration. The pinene does not react. The reaction may be applied directly to turpentine containing nopinene.
B. FULLMAN.

Manufacture of acetic anhydride. R. MEINGAST and M. MUDGAN, Assrs. to CONSORTIUM FÜR ELEKTROCHEM. IND. (U.S.P. 1,636,701, 26.7.27. Appl., 21.2.25. Conv., 25.2.24).—See E.P. 230,063; B., 1925, 568.

Production of aromatic aldehydes. A. E. CRAVER, Assr. to BARRETT Co. (U.S.P. 1,636,855, 26.7.27. Appl., 18.11.21).—See E.P. 189,107; B., 1923, 950 A.

Catalytic oxidation of aromatic hydrocarbons. A. E. CRAVER, Assr. to BARRETT Co. (U.S.P. 1,636,854, 26.7.27. Appl., 14.11.21).—See E.P. 189,091; B., 1924, 200.

Production of ethyl chloride. H. SUIDA (U.S.P. 1,637,972, 2.8.27. Appl., 3.2.25. Conv., 11.2.24).—See E.P. 229,298; B., 1925, 652.

Compounds of *p*-diamines and sulphur dioxide (E.P. 269,583).—See IV.

***N*-Alkyl-*p*-aminophenol** (E.P. 274,175).—See IV.

Hydrolysis of esters (E.P. 251,290).—See XII.

Condensation products (E.P. 263,853).—See XIV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Plate sensitivity and fogging agents: methylene-blue. J. SOUTHWORTH (Brit. J. Phot., 1927, **74**, 461—463).—A fine-grained, unwashed silver bromide emulsion, supposed to contain no nuclei, fogged on treatment with methylene-blue solution. Addition of thiocarbamides to such an emulsion (an alkaline emulsion) caused an increase in sensitiveness to light and also to methylene-blue, but the increase in the latter case was probably not proportional to the increase in light-sensitiveness. Desensitisation of an unwashed emulsion by a methylene-

blue-acridine-yellow solution does occur, but it is proportionately far less than with a rapid emulsion containing nuclei. A provisional working hypothesis of the action of methylene-blue is as follows:—The fogging action is dependent on the adsorption of methylene-blue to the grains. The desensitising effects are primarily due to adsorption of the dye by the nuclei. Protective dyes, such as acridine-yellow, function by preventing adsorption by the silver halide, but not by the nuclei. The sensitivity substance probably has a greater affinity for methylene-blue than for acridine-yellow. The fogging action of methylene-blue is ascribed to reduction of the dye by the developer to form its leuco-base, which in turn reduces the silver halide to form development nuclei of silver. Alternatively, fogging may be due to chemiluminescence accompanying reduction of the dye to its leuco-base, or else to some unstable fogging oxidation product of the developer. W. CLARK.

Halogen acceptor theory of sensitivity and the thioanilides. S. E. SHEPPARD and H. HUDSON (Phot. J., 1927, **67**, 359—361).—Thioanilides added to a standard emulsion made from relatively inert gelatin have an apparent desensitising effect, which is actually due to reduction of the densities in the toe of the characteristic curve, the % maximum density, and gamma. Fog due to white light is reduced, but the exposure scale is considerably altered. The thioanilides probably have the constitution HS·CR:NR′, where R′ is an aryl group and R is either an acyl or an aryl group. Complex formation with the silver halides probably takes place through the nitrogen atom, and not the sulphur atom. It appears that if the silver atom can be associated with the sulphur atom, silver sulphide is readily formed, but so long as the complex is anilided this is inhibited. The fact that sensitising is not effected by these compounds is in agreement that silver sulphide is not formed even in presence of alkali. The complex silver halide compound is a very effective halogen acceptor, but since it does not sensitise, the facts are contrary to the halogen acceptor theory of sensitising nuclei. The results are considered to confirm the view that silver sulphide is the effective sensitiser in the case of sensitisers containing sulphur, but they make of doubtful value any generalised halogen acceptor theory. Further, they offer a difficulty for the orientation theory of sensitising and latent image formation. W. CLARK.

Fogging action of persulphate. E. P. WIGHTMAN (Brit. J. Phot., 1927, **74**, 447—448).—It is confirmed that a dilute solution of bromine fogs a photographic plate. All high-speed emulsions contain a trace of free bromide. On treatment of the plate with oxidising desensitisers, free bromine would be liberated from this bromide, and would react slowly with the silver sulphide sensitivity-promoting specks to give metallic silver according to some modification of Hickman's hypothesis (cf. B., 1927, 126), or else to give silver bromide which remains for some time as silver and bromide ions. In either case it would lead to latent fog. W. CLARK.

Thermodynamic possibilities of the silver sulphide bromine-acceptor hypothesis of latent image formation. R. H. LAMBERT and E. P. WIGHTMAN (J. Physical Chem., 1927, **31**, 1249—1258).—The changes in free energy of the reactions concerned in Hickman's silver sulphide bromine-acceptor hypothesis (cf. B., 1927, 126), and in other reactions, have been calculated, and it is concluded that this hypothesis is thermodynamically sound, although certain of the intermediate reactions advanced by Hickman may be doubtful. It is further suggested that silver sulphide itself in the presence of silver bromide is photo-chemically decomposed, the bromide probably serving as an optical sensitiser, and as the sulphur acceptor giving rise to sulphur bromide and silver. The free energies of formation of silver sulphide and sulphur bromide, respectively, are — 6355 and — 3040 g.-cal. L. S. THEOBALD.

Photo-colorimeter. TOUSSAINT.—See I.

PATENTS.

Sensitised photographic papers. POLYGRAPHISCHE GES. (E.P. 250,267, 1.4.26. Conv., 3.4.25).—A photographic paper is coated with sensitive emulsion on both sides, and is also provided with at least one independent non-actinic layer between the two layers of emulsion. The non-actinic layer is decolorised during the photographic process, for example, during the development, or during fixing, washing, and drying, and the emulsion layers are strippable. W. CLARK.

Halation-free photographic plates. Z. Zelizy (G.P. 439,484, 5.4.25. Addn. to G.P. 393,762. Cf. B., 1924, 771).—Before coating with emulsion, both sides of the base are coated with a solution of transparent, chemically pure gelatin or collodion, to which is added a white powder which absorbs ultra-violet light (quinine hydrochloride, morphine, urea, etc.) in transparent aqueous or alcoholic solution. Further, a filter absorbing ultra-violet rays can be used in front of or behind the objective. W. CLARK.

Manufacture of kinematograph colour films. J. E. THORNTON (E.P. 272,986, 18.3.26).—A two-colour kinematograph film positive is produced by cementing together after printing two thin supports, one having an orange-red colloid relief image, the other a blue-green colloid relief image, the two films being cemented together with the image faces outwards. Two parallel rows of component images are printed on film material of double width, comprising a double-width support, carrying an insoluble substratum on which is coated an image-forming colloid layer, coloured before printing with two parallel stripes of suitable colours, and sensitised with dichromate. After printing, the pairs of differently-coloured images are developed and treated side by side on the double-width base to form a pair of relief images in complementary colours. W. CLARK.

[Tank] method of development of photographic film or the like and apparatus therefor. H. B. STRINGER and A. STRATTON (E.P. 275,349, 17.5.26).

Photographic films having lenticular elements. SOC. DU FILM EN COULEURS KELLER-DORIAN (E.P. 246,829, 19.1.26. Conv., 31.1.25).

Reproduction of photographic images on flms having lenticular elements. Soc. du Film en Couleurs Keller-Dorian (E.P. 245,118, 18.12.25. Conv., 27.12.24).

Compounds of *p*-diamines and sulphur dioxide (E.P. 269,583).—See IV.

XXII.—EXPLOSIVES; MATCHES.

Diethylene glycol dinitrate. Rinkenbach.—See XX.

Patent.

Combustible compositions for pyrotechnic, smoke- and flame-producing, and other like purposes. Vickers, Ltd., and E. E. F. Berger (E.P. 275,021, 9.8.26).—About 40 pts. by wt. of powdered calcium silicide are ground and mixed with 60 pts. by wt. of potassium nitrate to effect oxidation of the silicide without explosive effect, the mixture being compressed into thin cakes and granulated. The material may be associated as a priming or igniting agent with a mixture of powdered metal, a chlorinated hydrocarbon, and calcium silicide, by making granules of this mixture, coating these with the material by polishing or otherwise, and compressing. W. G. Carey.

XXIII.—SANITATION; WATER PURIFICATION.

Decomposition of cellulose in fresh sewage solids. H. Heukelekian (Ind. Eng. Chem., 1927, **19**, 928—930).—In studies of the rate of decomposition of cellulose during digestion of limed and unlimed unseeded fresh sewage solids, in which the cellulose contents of the sludges were determined from time to time by use of Schweitzer's reagent, about 36% of the cellulose disappeared in the first week and 79% in 3 weeks in the unlimed material, as compared with 73% in the first week and 96% in 3 weeks in the case of limed mixtures. Lime, by neutralising the acid decomposition products, accelerates also the rate of decomposition of the non-cellulosic organic matter. W. T. Lockett.

Determination of organic matter in sewage and effluent. W. E. Abbott (Ind. Eng. Chem., 1927, **19**, 919—921).—A study of Adeney's dichromate method for the determination of organic matter in sewage and effluent, more particularly with regard to the conditions required for successful chloride removal, led to the adoption of the following procedure: 25 c.c. of a raw sewage or an equivalent volume of other liquid are placed in a small evaporating basin, 0·3 c.c. of sulphuric acid is added, and the liquid evaporated with occasional stirring until about 0·7 c.c. remains, when the residue is vigorously stirred at intervals during 3 or 4 min. to remove the chloride; 25 c.c. of 0·125*N*-dichromate solution are added, and evaporation is resumed until about 1 c.c. remains; 5—10 c.c. of sulphuric acid are added and the basin is left on the water bath for 2 hrs.; the liquid is then diluted, and the residual dichromate titrated with ferrous sulphate, using potassium ferricyanide as external indicator. The liquid in the basins undergoing digestion should be maintained above 94°, and blank determinations are required. Although the oxygen absorbed from dichromate by various organic substances when subjected to the modified test varied from 60 to 99% of the oxygen calculated as necessary to convert them into carbon dioxide, water, and ammonia, the dissolved oxygen absorption (until nitrification commenced) of raw sewage (freed from suspended matter) indicated that the dichromate absorption approximates to the oxygen required to convert all the organic matter present in sewage into carbon dioxide, water, and ammonia. No definite relationship was established between the dichromate absorption and the 5 days' dissolved oxygen absorption. With crude sewage the dichromate absorption is on the average 5·1 times as great as the permanganate absorption (A.P.H.A. 1923 method); with sewage free from suspended matter 4·5 times; and for well purified activated sludge effluents 2·5 times—results which indicate that the dichromate absorption affords a more sensitive index of the course of purification. W. T. Lockett.

Repellants for blowflies. R. C. Roark, D. C. Parman, F. C. Bishopp, and E. W. Laake (Ind. Eng. Chem., 1927, **19**, 942—943).—Observations as to the number of flies visiting fresh beef liver treated with various repellants and the number visiting untreated fresh beef liver indicated that the most effective repellants are materials, *e.g.*, copper carbonate, which absorb, adsorb, or inhibit the formation of the volatile compounds evolved by decomposing meat which attract the flies to the meat. In addition to various copper compounds, such strong antiseptics as mercuric chloride, potassium permanganate, sodium salicylate, etc. when applied to meat render it almost entirely non-attractive. Some strongly odorous materials are quite effective when undiluted, but lose nearly all their efficacy or become actually attractive when diluted 1 to 9 with an inert vehicle. W. T. Lockett.

Lubricating oils as insecticides. Green.—See XVI.

Patents.

Disinfectant tablets, bricks, etc. British Dyestuffs Corp., Ltd., T. H. Fairbrother, and A. Renshaw (E.P. 274,187, 14.4.26).—Mixtures of auramine with plaster of Paris, diatomite, or a solid volatile medium, with or without the addition of sodium chloride, are moulded into various shapes and suspended in the water of a flush cistern. The auramine diffuses slowly and regularly and confers disinfectant properties to the water. C. Ranken.

Preparation of water-softening materials. G. W. Prather (U.S.P. 1,636,942, 26.7.27. Appl., 10.8.16).—A mineral having a base of hydrated aluminium silicate is dried until practically all the uncombined moisture is driven off and, after being broken to a suitable size, is baked at a high temperature with sodium hydroxide until the latter fuses and permeates the mineral, which is finally washed free of excess of soda. W. G. Carey.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCTOBER 14, 1927.

I.—GENERAL; PLANT; MACHINERY.

Recovery of volatile solvents in chemical industries. Bodewig (Z. ges. Schiess- u. Sprengstoffw., 1927, **22**, 167—169).—The volatile solvent recovery plant of the Cheminova Co. of Berlin is described. The method is in essence the same as that of the Brégeat recovery process, and consists in absorbing the solvent vapours in certain cresols which form with them complex compounds. These compounds are easily decomposed on heating, by which means the solvents are obtained in a pure state. The plant consists of three parts in which absorption, decomposition, and rectification, respectively, take place. Absorption of the vapours from the vapour-air mixture is effected by passing the mixture up a tower filled with iron spirals in counter-current to a stream of the cresol absorbent. The cresol–solvent mixture then passes to the decomposer, where it is heated, yielding up the solvent, which passes to the rectification plant. The cresol from the decomposer is then available for use again in the absorber. The application of the process to various industries is outlined. The recovery of ether–alcohol in a Chardonnet silk plant by this means was over 90% and of acetone in an artificial silk factory was 95—98%. S. Binning.

Testing machine for repeated impact. Smith and Warnock.—See X.

Patents.

Dry kiln. C. G. Ullin (U.S.P. 1,637,166, 26.7.27. Appl., 18.8.26).—The kiln is provided with air inlet and outlet conduits spaced apart and with pipe connexions between them, the air being circulated by a blower. Means are provided for moistening the air in the kiln and for separating moisture from the air passing through the pipe connexions, such moisture being delivered through an exhaust opening into the atmosphere. H. Holmes.

Retort. K. L. Ford, Assr. to Glass Container Assoc. of America (U.S.P. 1,636,768, 26.7.27. Appl., 12.3.26).—A closed vessel is provided with an intake conduit for heating vapour and an exhaust conduit communicating with the vessel at several points remote from the inlet. To ensure uniform heating throughout the vessel the gaseous contents are exhausted from and returned to it under the aspirating action of the inflowing vapour. H. Holmes.

Preventing corrosion in condensers. Internat. General Electric Co., Inc., Assees. of Allgem. Elektricitäts-Ges. (E.P. 258,908, 28.9.26. Conv., 28.9.25).—In a condenser for a steam power plant the surfaces in contact with the cooling water are protected from electrolytic erosion by a coating of "Bakelite," which is preferably applied in the liquid condition and solidified by heating under pressure. H. Holmes.

Ball or tube mill. W. Köppen and C. Pfeiffer (U.S.P. 1,636,138, 19.7.27. Appl., 8.12.26. Conv., 8.12.25).—A preliminary grinding chamber communicates with a fine-grinding chamber through an intermediate chamber, the wall between the first two being formed of concentric rings provided with an entirely free peripheral passage for the material to be ground. H. Holmes.

Ball mill. W. M. Barker (U.S.P. 1,636,585, 19.7.27. Appl., 13.10.24).—One end of a rotary grinding drum is provided with a short trunnion mounted in a bearing bracket, the other end having an inlet for the material to be ground. A removable blower casing, surrounding the drum and communicating with it, has separate outlets for the material and for the air. H. Holmes.

Pulverising machine. W. K. Cruickshank (U.S.P. 1,633,164, 21.6.27. Appl., 28.9.26).—A pulverising apparatus is fitted with rotary and stationary cutters, and with a heating jacket, being adapted to pulverise sodium sulphate decahydrate and convert it into the octahydrate. T. S. Wheeler.

Rotary dryer. C. Miller (U.S.P. 1,637,831, 2.8.27. Appl., 7.3.25).—In an upright cylindrical chamber rotating about its vertical axis, an air entry at the bottom of the chamber wall faces the direction of rotation and an air exit at the top the reverse direction. C. A. King.

[Centrifugal] separation of liquids from solids. C. R. Altenhein. From W. Klein (E.P. 274,958, 28.4.26).—To enable a screen of coarser mesh and thicker metal to be used, the material to be drained is first graded and the different grades are fed separately, but simultaneously, into the machine so that coarser material impinges directly on the screen to form thereon a porous filtering layer for the finer material. The grades are thrown towards the screen in proper rotation by a feed table with projecting vanes. The draining of the finer material is facilitated when the bulk of the material is in the coarser grades. When a conical screen with slot perforations is used, the length of each slot is arranged oblique to the direction of travel of the material to facilitate the passage of the material over the screen surface. The sludge which falls when the machine is retarded suddenly is caught in a cup provided with draining channels. H. Holmes.

Apparatus for the [catalytic] hydrogenation of liquids and semi-liquids. G. R. Schueler (E.P. 274,952, 28.4.26).—In a catalytic unit as described in E.P. 273,045 (B., 1927, 690) each disc may be of woven

wire, or of perforated sheet metal, or of a spiral strip of the metal with distance pieces between the convolutions, and may be provided with spacing corrugations, ribs, or projections. A uniform oxidation of the unit is effected by dismantling the discs and suspending them alternately with cathode plates in an electrolytic bath.

H. HOLMES.

Process and apparatus for the continuous beating of viscid liquids. D. G. STEELY (E.P. 274,976, 1.5.26).—The liquid is agitated in a large volume of air with provision for accommodating the continuously increasing bulk of the aerated material passing through the beating chamber to an unrestricted outlet. The chamber is preferably a horizontal cylinder provided with a longitudinal rotary shaft carrying the beating and propelling devices, the shaft bearings being remote from the beating zone, and blades for forcing air through the cylinder are carried by the shaft at the inlet end. A constant head of liquid is introduced between these blades and the beating zone through one or more openings provided with valves. The beating devices are more widely spaced toward the discharge end, *e.g.*, an open helical coil of increasing pitch may be used. H. HOLMES.

[Reflux] extraction method and apparatus. H. BUEL, Assr. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,636,550, 19.7.27. Appl., 9.7.25).—Extractives injuriously affected by high temperatures are obtained by the use of a reflux extractor operating under reduced pressure. The material is placed in a container to which an exhaust pump is connected through a reflux condenser, and the solvent is vaporised in a vessel connected to the container by a vapour supply duct and a return duct for the solvent and extractive.

H. HOLMES.

Determination of equilibrium vaporisation end-points. C. E. PARSONS and R. STEVENSON, Assrs. to W. P. DEPPE (U.S.P. 1,632,748, 14.6.27. Appl., 6.10.26).—The liquid under examination is completely vaporised by passage down a heated column, the vapour being led into a chamber containing a glass tube coated at one end with platinum black. Means are provided for cooling the coated tip by a current of air, and for observing the temperature at which condensation occurs on it.

T. S. WHEELER.

Apparatus for bringing liquids and gases into intimate contact. P. F. HOLMES, D. M. HENSHAW, and W. C. HOLMES & CO., LTD. (E.P. 275,092, 7.2.27).—Apparatus as described in E.P. 5113 of 1895 and 162,166 (B., 1896, 340; 1921, 422 A) is modified to cause the gases to travel through all the brushes in the same direction to or from the shaft. Still chambers may be located between adjacent compartments, and may serve for the passage of the gases from a point near the shaft in one compartment to a point remote from the shaft in the next compartment, or in the reverse direction.

H. HOLMES.

Production of a catalyst carrier. C. R. DOWNS (U.S.P. 1,636,685, 26.7.27. Appl., 9.12.26).—A carrier for use in the catalytic oxidation of organic compounds in the vapour phase comprises iron particles coated with aluminium. T. S. WHEELER.

Imparting to drying means a large superficial area. E. SMOLCZYK (U.S.P. 1,633,959, 28.6.27. Appl., 3.6.26. Conv., 8.6.25).—Phosphorus pentoxide can be deposited on pumice by first impregnating the latter with phosphoric acid. T. S. WHEELER.

Manufacture of a filter aid. H. S. THATCHER, Assr. to CELITE CO. (U.S.P. 1,632,458, 14.6.27. Appl., 29.7.25).—Diatomaceous earth is ground so that not more than 10% remains on 150-mesh and 15—50% of the particles settle in distilled water at 20° at about 0·36 cm./min. T. S. WHEELER.

Filters for purifying and sterilising air and gases. M. GRÄFIN VON LINDEN, R. MELDAU, and DEUTS. LUFTFILTER BAUGES. M.B.H. (E.P. 274,942, 23.4.26).—A filtering material composed of or containing copper is coated with a viscous solution of a copper salt such as a mixture of paraffin oil and copper oleate. The solution may be applied to copper plates or sieves, or may be placed with finely-divided copper in a container. The nature and proportions of the oil and salt used may be such that the mixture is destructive to certain bacteria and innocuous to others. H. HOLMES.

Gas analysis apparatus. K. L. TATE, Assr. to TAYLOR INSTRUMENT COS. (U.S.P. 1,633,352, 21.6.27. Appl., 23.2.26).—A current of a mixture of gases, *e.g.*, flue gas, is drawn through a capillary tube terminating in a chamber with a restricted outlet. The current enters the capillary tube at atmospheric pressure, and leaves the chamber at a constant rate, the restricted outlet being subject to the conditions of maximum flow. Means are provided for compensating for changes in atmospheric pressure, and for determining the pressure in the chamber, which varies as the viscosity of the gas under examination, and hence, in this instance, with its carbon dioxide content. T. S. WHEELER.

Edge [stream-line] filter. E. A. FORSBERG, Assr. to DE LAVAL SEPARATOR CO. (U.S.P. 1,640,698, 30.8.27. Appl., 4.9.24. Conv., 18.9.23).—See E.P. 226,274; B., 1925, 157.

Machine for separating solids from liquids. W. C. LAUGHLIN, Assr. to LAUGHLIN FILTER CORP. (U.S.P. 1,640,707, 30.8.27. Appl., 29.9.23).—See E.P. 235,916; B., 1926, 255.

Method and apparatus for drying or conditioning products composed of different components having different moisture contents. E. C. R. MARKS. From INDUSTRIAL DRYER CORP. (E.P. 275,681, 15.2.26).—See U.S.P. 1,562,764; B., 1926, 81.

Apparatus for analysing [furnace] gases. J. ALSBERG and P. J. RALPH, Assr. to J. ALSBERG (U.S.P. 1,633,881, 28.6.27. Appl., 5.2.24). — See U.S.P. 1,485,757; B., 1924, 496.

[Compression] refrigerating machine. A. A. KUCHER (E.P. 261,709, 13.8.26. Conv., 19.11.25).

Transverse-current cooling tower. M. HAMON (E.P. 263,114, 18.11.26. Conv., 16.12.25).

[Automatic] siphon. N. SWINDIN (E.P. 275,315, 3.12.26).

Apparatus for determining, indicating, and controlling the moisture content of paper and like materials. J. D'A. CLARK (E.P. 275,741, 18.5. and 13.7.26).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Peat. II. Distillation under reduced pressure of certain constituents of peat. J. Reilly and (Miss) J. Sullivan (Sci. Proc. Roy. Dubl. Soc., 1927, **18**, 383—388; cf. B., 1926, 906).—In order to study the decomposition of peat, the authors separated the material into fractions. The peat was first extracted with dilute hydrochloric acid, the amount of extract thus obtained being 5·4% of the dry peat. The residue was separated by extraction with 4% ammonia solution into "insoluble humin" (67%) and "soluble humin" (27·6%). The "humus" precipitated from the "soluble humin" by hydrochloric acid was further fractionated into "alcohol extract of humus" (7%) and "humic acid" (15·5%). Each of these fractions was fractionated under reduced pressure, the products being collected in two receivers—one kept at a constant temperature of 60° and the other in a freezing mixture. The products were coke, 60° distillate, 0° distillate, and gas. Humic acid and hydrochloric acid extract gave very little tar. The highest percentage of coke was obtained from the humic acid, and the lowest from the alcoholic extract of humus. The greatest volume of gas was obtained from the insoluble humin. The gases consisted principally of carbon dioxide, with smaller amounts of carbon monoxide and hydrocarbons. G. W. Robinson.

Conditions for success of low-temperature carbonisation. W. E. Davies (Chem. Met. Eng., 1927, **34**, 480—483).—Mainly in the sphere of economics. The importance of throughput is stressed, and the most promising line of development is considered to be carbonisation at a gas-works in continuous vertical retorts, with a feed so adjusted that low-temperature conditions are approached, the loss in gaseous therms being compensated by steaming. It is also emphasised that avoidance of cracking of the tar and oil produced not only increases the yield of distillate, but results in heat economy. The time required for carbonisation may be almost halved. C. Irwin.

Apparatus for determining the softening point of paraffins, waxes, pitches, asphalts, etc. H. Herbst (Petroleum, 1927, **23**, 1079—1080).—See B., 1927, 244.

Coke for blast-furnace working. Gill.—See X.

Patents.

Combination [coke] oven. J. Becker, Assr. to Koppers Co. (U.S.P. 1,623,460, 5.4.27. Appl., 19.8.25).—Independent channels suitably proportioned for admitting air or gas to the flues of an oven are provided so that the oven can be operated either with preheated gas and preheated air or with cold rich gas and preheated air, with complete utilisation of all the regenerators. S. Pexton.

Coking still. R. Cross, Assr. to Gasoline Products Co. (U.S.P. 1,622,573, 29.3.27. Appl., 12.4.24).—A horizontal cylindrical still rotates around a fixed central shaft carrying a drum. The still is externally heated and the petroleum coke is scraped from the still walls by the rotation of the still around the fixed drum. S. Pexton.

Wood charcoal briquettes. M. L. Y. de Paniagua (F.P. 615,149, 14.9.25).—Powdered charcoal is mixed with an organic binder, *e.g.*, tar, preferably with the addition of a dichromate, compressed, and carbonised above 350°. A. B. Manning.

Manufacture of decolorising carbon. L. Wickenden and S. A. W. Okell, Assrs. to Industrial Chemical Co. (U.S.P. 1,634,477—8 and 1,634,480, 5.7.27. Appl., [A] 16.6.21; [B] 18.2.22; [C] 22.3.23).—(A) Carbonised vegetable material, *e.g.*, from waste sodium sulphite liquors, is heated in a rotary kiln at 900°, and then passed against a current of steam and carbon dioxide down an electric shaft-furnace maintained at 900°. (B) The electric furnace contains a lower rotating electrode, through which the gas current is introduced. The material when sufficiently disintegrated is removed by the gas current. (C) Material treated as in (B) is heated with air at 400° in a rotary kiln. T. S. Wheeler.

Electric furnace for revivifying decolorising carbon etc. L. Wickenden and W. F. Demmerle, Assrs. to Industrial Chemical Co. (U.S.P. 1,634,479, 5.7.27. Appl., 3.7.22).—The furnace comprises a cylinder rotating around a vertical axis, and containing stationary electrodes which also serve as agitators. T. S. Wheeler.

Production of metallic carbon. C. Robinson, Assr. to Westinghouse Electric & Manuf. Co. (U.S.P. 1,637,052, 26.7.27. Appl., 1.8.21. Renewed 15.12.25).—A mixture of anthracite coal and cellulose is heated in an electric furnace at 3500° to yield pure elementary carbon in a porous form, which on fusion forms a malleable ductile product of value in the manufacture of electric lamp filaments. T. S. Wheeler.

Production of highly active carbon. I. G. Farbenind. A.-G., Assees. of S. Münch (G.P. 439,114, 14.3.23. Addn. to G.P. 438,670).—Wood or other cellulosic material impregnated with a solution containing sodium and ammonium sulphates is calcined above 500°, and the product is extracted with water. L. A. Coles.

Production of carbon black. L. J. Dales (U.S.P. 1,633,071, 21.6.27. Appl., 4.4.21. Renewed 27.8.24).—Natural gas is passed through a chamber in which it is heated, the issuing gas then interchanging heat with the entering gas. Any liquid condensate is removed, and the residual gas is burned under the heating chamber to form carbon black. T. S. Wheeler.

Production of carbonaceous material. A. Oberle (U.S.P. 1,632,845, 21.6.27. Appl., 31.7.24).—Carbonaceous residues from cracking mineral oil are heated at about 540° in a current of steam, first under increased and then under reduced pressure, being finally treated with a mixture of nitric and sulphuric acids. T. S Wheeler.

Production of active charcoal from cacao refuse. E. Tiede (G.P. 440,381, 3.4.25).—The refuse is carbonised at red heat after removal of fat and of theobromine by sublimation. L. A. Coles.

Gaseous fuel [for welding etc.]. J. R. Rose, Assr. to Carbo-Hydrogen Co. of America (U.S.P. 1,628,067, 10.5.27. Appl., 31.12.19).—A mixture containing 80—98% H_2 and 20—2% CH_4, together with a

small proportion of propane and butane, is of use as a fuel for welding and cutting metal. S. Pexton.

Manufacture of gas from coal and like carbonaceous materials. M. W. Travers and F. W. Clark (E.P. 267,724, 7.4.26. Addn. to E.P. 198,777; B., 1923, 759 a).—In the process of completely gasifying coal in an elongated gas producer, as described in the prior patent, a core of uncarbonised coal, into which the hot gases do not penetrate, is formed. By introducing a mechanical agitator into the distillation chamber, the plastic layer of coal surrounding this core may be broken down. S. Pexton.

Fuel gas distributing systems for coke ovens. Koppers Co., Assees. of J. Becker (E.P. 252,676, 20.3.26. Conv., 29.5.25).—When fuel gas is supplied to the burners of a coke oven through a horizontal duct at the top of the regenerator walls the fuel gas increases in temperature and velocity towards the centre of the oven. In order to regulate the amounts of gas fed to individual flues, a second gas-distributing flue is built below the sole of the regenerators, and is connected to the upper distributing flue by vertical ducts. The regulating orifices may be placed at the base of these vertical ducts where the temperatures are uniformly low. S. Pexton.

Gas production from powdered charcoal. C. Hilker (G.P. 439,011, 14.1.23).—The powdered charcoal, mixed with a suitable slag-forming flux, is charged, without admixed air, in at the top of a vertical shaft in such a way that it is distributed over the cross-section of the shaft by centrifugal action, and is gasified in a current of air entering at the bottom of the shaft. A. B. Manning.

Water-gas generator for bituminous fuel. O. Misch (E.P. 255,447, 12.7.26. Conv., 16.7.25).—Bituminous fuel is distilled in an externally heated retort superimposed upon a water-gas generator in which the carbonised fuel is then gasified. During the "blow" period the "blow" gases are burnt in the spiral combustion chamber surrounding the retort, whilst during the "run" part of the water-gas is withdrawn from the lower part of the generator and burnt around the retort, the remainder passing through the distillation retort, and thus assisting the carbonisation. S. Pexton.

Manufacture of water-gas [from powdered fuel]. I. G. Farbenind. A.-G., Assees. of F. Winkler (G.P. 438,843, 26.7.24. Addn. to G.P. 437,970; B., 1927, 548).—The procedure of the main patent is modified by passing the steam downwards through the incandescent material, so that during the steaming period the material is quiescent. The gas produced is then free from dust. A. B. Manning.

Purification of [coal] gas. W. H. Fulweiler and C. W. Jordan, Assrs. to U.G.I. Contracting Co. (U.S.P. 1,632,758, 14.6.27. Appl., 24.6.26).—Sodium carbonate solution which has been used to remove sulphur from coal gas is revivified by being stirred with an immersed paddle, under which a stream of minute bubbles of air is introduced. The sulphur separating is removed at the surface. T. S. Wheeler.

Gas-purifying apparatus [oxide purifier]. T. W Stone, Assr. to Western Gas Construction Co. (U.S.P 1,632,251, 14.6.27. Appl., 16.2.22).—The purifying material is packed in upper and lower tiers, valves being provided to enable the gas to be passed in either direction through the tiers, to permit complete utilisation of the purifying material. T. S. Wheeler.

Gas-separating apparatus. M. M. Reynolds (U.S.P. 1,623,636, 5.4.27. Appl., 14.10.25).—Gas to be purified from dust is streamed through a compartment containing inclined deflectors and screened openings. The deflectors, arranged in advance of each screened opening, baffle the gas and serve to deposit part of the dust on the bottom of the chamber, the remainder being removed by the screens. S. Pexton.

Separation of carbon dioxide from gas mixtures by means of ammoniacal liquor. Bamag-Meguin A.-G. (G.P. 440,324, 28.8.25).—The gases and ammoniacal liquor pass in the same direction through a series of scrubbers, cooling apparatus being provided between each pair of scrubbers to remove the heat of dissolution and neutralisation of the carbon dioxide.
L. A. Coles.

Apparatus for the distillation of solid bituminous materials. J. Daniels (G.P. 440,169, 30.6.25).—Coal is carbonised in radial chambers on the outer jacket of a rotary retort; before or during its introduction into the carbonising chamber the coal is compressed, the compression chamber being closed from the supply chamber during the operation by a damper. A dense, homogeneous carbonisation product is obtained.
A. B. Manning.

Distillation of bituminiferous material. J. J. Jakowsky (U.S.P. 1,622,722, 29.3.27. Appl., 11.1.26).—Solid bituminous material is exposed to a heat-radiating surface so that only the upper surface of the mass is heated at the desired temperature. The mass is progressively stirred so as to expose it gradually to surface distillation. S. Pexton.

Conversion [cracking] of the components of petroleum and the like into more volatile products. W. C. and F. E. Wells (U.S.P. 1,635,519, 12.7.27. Appl., 10.8.20).—Petroleum is introduced at the bottom of a still containing molten lead at about 540°, a continuous flow of the higher fractions of the liquid being maintained across the surface of the metal to remove carbonaceous impurities. T. S. Wheeler.

Production of benzine, kerosene, and higher hydrocarbons from crude petroleum oil emulsions of the water-in-oil type. P. J. de Kadt (Dutch P. 15,167, 7.4.24).—The emulsion is converted by the addition of a suitable agent, *e.g.*, resin soap, into an intermediate stage which cannot be characterised as either a water-in-oil or an oil-in-water emulsion, and, without separation of the water, is then distilled. No frothing or bumping occurs during distillation. A. B. Manning.

Production of light hydrocarbon liquids. E. C. Herthel, Assr. to Sinclair Refining Co. (U.S.P. 1,632,967, 21.6.27. Appl., 4.4.25).—Heavy oil is heated at about 1000° and 9 atm. pressure, the distillate being passed up a column in counter-current to a stream of fresh oil, which is added at about twice the rate at which the distillate is withdrawn. T. S. Wheeler.

Refining mineral oils etc. with liquid sulphur dioxide. ALLGEM. GES. FÜR CHEM. IND. M.B.H. (F.P. 615,553, 3.5.26. Conv., 5.3.26).—Before the solution containing the sulphur dioxide extract enters the evaporator working under the condenser pressure, it passes through one or more evaporators working under higher pressures, only one of which—that working under the highest pressure—is heated from an external source. Each of the others is heated by acting as a condenser for the evaporator working at the next higher pressure.
A. B. MANNING.

Art of refining oils. C. LEAVER, Assr. to IMPERIAL OIL, LTD. (U.S.P. 1,635,718, 12.7.27. Appl., 11.3.21. Conv., 13.8.20).—The oil is mixed with partially-spent sulphuric acid in a pipe fitted with baffles and separated from the sludge in a settling tank, being then treated with fresh acid and recovered from the resulting partially spent acid in a similar unit. The same process can be used for the alkaline washing. T. S. WHEELER.

Prevention of acidity in [transformer] oils. C. J. RODMAN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,633,956, 28.6.27. Appl., 12.3.25). —The formation of acid in transformer oil is inhibited by coating immersed copper parts with tin.
T. S. WHEELER.

Preparation of an adsorbent for [liquid petroleum] oils. W. D. RIAL and E. W. GARD (U.S.P. 1,634,514, 5.7.27. Appl., 8.9.25).—Adsorbent clay is treated at 190° with pure petroleum distillate containing sulphuric acid, until adsorption of the latter is complete, and is then separated and freed from adherent oil in a centrifuge. T. S. WHEELER.

Revivification of spent adsorbents for oil treatments. P. W. PRUTZMAN, Assr. to CONTACT FILTRATION CO. (U.S.P. 1,633,871, 28.6.27. Appl., 2.9.22).—The adsorbents are washed first with light oil, then with a mixture of benzene and alcohol, and finally heated with water. T. S. WHEELER.

Petroleum oil composition. A. A. SOMERVILLE, Assr. to R. T. VANDERBILT CO., INC. (U.S.P. 1,634,054, 28.6.27. Appl., 19.6.26).—*p*-Aminophenol (0·5—1%) is added to petroleum insulating and lubricating oils to inhibit oxidation at high temperatures.
T. S. WHEELER.

Method of using low-compression fuels. C. F. KETTERING and T. MIDGLEY, JUN., Assrs. to GENERAL MOTORS CORP. (U.S.P. 1,635,216, 12.7.27. Appl., 3.1.24).—Ethyl iodide is employed as an anti-knock material. T. S. WHEELER.

Coking retort oven. J. VAN ACKEREN, Assr. to KOPPERS CO. (U.S.P. 1,633,911, 28.6.27. Appl., 18.1.21).—See E.P. 230,167; B., 1925, 345.

Process of distilling oil in presence of comminuted carbonaceous fuel. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,641,305, 6.9.27. Appl., 4.11.20).—See E.P. 171,075; B., 1922, 701 A.

Process and apparatus for cracking hydrocarbon oils. W. F. FARAGHER and W. A. GRUSE, Assrs. to GULF REFINING CO. (U.S.P. 1,640,444, 30.8.27. Appl., 15.1.21). —See E.P. 174,085; B., 1923, 301 A.

Self-sealing door for coke ovens. H. SCHWARZ (E.P. 268,313, 3.3.27. Conv., 26.3.26).

Gas analysis apparatus (U.S.P. 1,633,352).—See I.

Asphaltic paving (U.S.P. 1,634,241). **Bituminous concrete** (E.P. 275,364).—See IX.

IV.—DYESTUFFS AND INTERMEDIATES.

Identification of vat dyes. VAIS.—See VI.

PATENTS.

Manufacture of an azo dye. H. WAGNER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,635,594, 12.7.27. Appl., 22.12.25. Conv., 11.6.23).—Diazotised or tetrazotised aminoazo or diaminoazo dyes, containing at least one free hydroxyl or carboxyl but no sulphonic group, are coupled with 1 or 2 mols. of a 2 : 3-hydroxynaphthoic arylamide to give *products* yielding fast black shades on cotton. *E.g.*, diazotised *p*-phenylenediamine is coupled with β-naphthol, and the diazotised product is coupled with 2 : 3-hydroxynaphthoic 5-chloro-2-toluidide.
T. S. WHEELER.

Manufacture of mordant [azo] dyes. DURAND & HUGUENIN A.-G. (E.P. 255,086, 8.7.26. Conv. 13.7.25).— *p*-Phenylenediaminesulphonic acid is monoacetylated, diazotised, and coupled with an *o*-hydroxy-acid, and the product is hydrolysed, rediazotised, and coupled with the same or a different *o*-hydroxy-acid. Similar products are obtained from *p*-nitroanilinesulphonic acids, diazotised, coupled, reduced, rediazotised, and coupled. Chrome-mordanted wool is dyed in reddish-orange shades.
C. HOLLINS.

Manufacture of disazo [mordant] dyes. I. G. FARBENIND. A.-G., Assees. of FARBENFABR. VORM. F. BAYER & CO. (E.P. 251,637, 28.4.26. Conv., 28.4.25).— An aminobenzoic acid, containing a second carboxyl group or a sulphonic group or hydroxyl and sulphonic groups, is diazotised and coupled with α-naphthylamine-6- or -7-sulphonic acid, the product rediazotised and coupled with an aromatic *o*-hydroxycarboxylic acid, to give chrome-browns for wool or for printing on cotton, which are fast to chlorine. Examples are 4-aminophthalic acid → α-naphthylamine-6-sulphonic acid → *m*-cresotic acid; 3-amino-5-sulphosalicylic acid → α naphthylamine-6-sulphonic acid → salicylic acid.
C. HOLLINS.

Anthraquinone dyes and dyeing therewith. BRITISH DYESTUFFS CORP., LTD., A. SHEPHERDSON, and W. W. TATUM (E.P. 275,421, 19.8.26).—2-Halogeno-4-(sulphoarylamino)-1-hydroxyanthraquinones are boiled with aqueous sodium sulphite, filtered from unchanged monosulphonic acid, and evaporated to dryness. The product from 2-chloro-4-(sulpho-*p*-tolylamino)-1-hydroxyanthraquinone dyes wool from an acid bath a red-blue, becoming green by after-chroming. C. HOLLINS.

Production of anthraquinone. A. E. CRAVER, Assr. to BARRETT CO. (U.S.P. 1,636,856, 26.7.27. Appl., 5.12.21).—A mixture of anthracene vapour and air (15 pts.) is passed at 475° over a catalyst comprising uranium oxide (89%) and molybdenum oxide (11%), the time of contact being about ½ sec. T. S. WHEELER.

Manufacture of *N*-dihydro-1 : 2 : 1′ : 2′-anthraquinoneazine. L. H. ULICH and R. J. GOODRICH, Assrs. to NEWPORT CO. (U.S.P. 1,634,473, 5.7.27. Appl., 4.4.25. Cf. U.S.P. 1,580,700; B., 1926, 577).—Dibromo-*N*-dihydro-1 : 2 : 1′ : 2′-anthraquinoneazine is heated,

with or without addition of solvents, diluents, or catalysts, with 33% alcoholic potassium hydroxide solution (6 pts.) for 1 hr. T. S. WHEELER.

Manufacture of azine dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 275,301, 29.4.26).—1 : 8-Naphthasultam-3-sulphonic or -3 : 6-disulphonic acid, when heated with a primary or secondary arylamine at 110—160° preferably in presence of a salt of the arylamine, exchanges the 3-sulphonic group for an arylamino-group. The products condense with nitrosodimethylaniline etc. (or may be oxidised with *p*-diamines) to form violet azine acid dyes for wool. 1 : 8-Naphthasultam-3-sulphonic acid gives with aniline 3-*anilino*-1 : 8-*naphthasultam*, m.p. 168—170°. 1 : 8-Naphthasultam-3 : 6-disulphonic acid with aniline gives 3-*anilino*-1 : 8-*naphthasultam*-6-*sulphonic acid* (*aniline* salt, m.p. 277—278°), and similar *compounds* are formed from *p*-toluidine and *p*-anisidine. C. HOLLINS.

Manufacture of dyes [*iso*violanthrone]. BRITISH ALIZARINE CO., LTD., W. H. DAWSON, and P. BEGHIN (E.P. 275,283, 4.3.26).—*iso*Violanthrone [*iso*dibenzanthrone] is obtained by heating 3-halogenobenzanthrones, having a free 2-position, with 8—10 pts. of aniline (or other arylamine) and 4—5 pts. of sodium or potassium hydroxide at 150—185°. Impurities are extracted from the dried product by means of aniline. C. HOLLINS.

Manufacture of thioindigo vat dyes. K. THIESS, C. J. MÜLLER, E. RUNNE, and A. SCHAEFFER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,632,252, 14.6.27. Appl., 22.6.26. Conv., 27.6.25).—3 : 3′ : 5 : 5′-Tetramethyl-, 3 : 3′-dihalogeno-5 : 5′-dimethyl-, 5 : 5′-dihalogeno-3 : 3′-dimethyl-, and 3 : 3′ : 5 : 5′-tetrahalogeno-thioindigos can be halogenated to the corresponding 4 : 4′-dihalogeno-derivatives by treatment with a halogen in presence of a catalyst, *e.g.*, antimony pentachloride, and a diluent, *e.g.*, chlorosulphonic acid. The products give on cotton in the vat reddish violet shades, fast to boiling. The 3 : 3′-dimethyl compounds are not claimed as new. T. S. WHEELER.

Manufacture of indigoid dyes. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 262,457, 3.12.26. Conv., 4.12.25).—Tetrahalogenated thioindoxyls are condensed with an isatin, a thioisatin, or with halides or α-anils of these. 4 : 5 : 6 : 7-Tetrachlorothioindoxyl with 5 : 7-dichloroisatin chloride gives a violet vat dye, with 5 : 7-dichloroisatin a brown, with the *p*-dimethylaminoanil of 9-bromo-ββ-naphthathioisatin a violet. C. HOLLINS.

Manufacture of products [indigosols] for dyeing or printing textile fibres and other materials. DURAND AND HUGUENIN SOC. ANON., Assees. of I. G. FARBENIND. A.-G. (E.P. 267,952, 17.3.27. Conv., 17.3.26. Addn. to E.P. 186,057 ; B., 1924, 252).—The indigosols of the prior patent are obtainable as ammonium salts by treating a leuco-vat dye with aminosulphonic acid in presence of a tertiary base (pyridine, dimethylaniline) and preferably in suspension in an inert medium (chlorobenzene) at 75—85°. C. HOLLINS.

Production of benzidine and derivatives. R. A. NELSON, Assr. to NATIONAL ANILINE & CHEMICAL CO., INC. (U.S.P. 1,633,123, 21.6.27. Appl., 22.6.20).—Hydrazobenzene in benzene solution is treated at 10° with concentrated hydrochloric acid to separate benzidine dihydrochloride in satisfactory yield. T. S. WHEELER.

Manufacture of wool and leather dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (E.P. 274,999, 21.6.26).—A halogenated aromatic nitro-compound containing reactive halogen (other than 1-halogeno-2 : 4-dinitrobenzene ; cf. E.P. 13,672 of 1912 ; B., 1912, 1074) is condensed with 4-aminodiphenylamine or a derivative thereof. The products, which must contain at least two nitro-groups and a sulphonic group, dye wool and leather in fast yellow to brown shades. 4-Nitro-4′-aminodiphenylamine-2-sulphonic acid is condensed with *o*- or *p*-chloronitrobenzene or 4-chloro-3-nitrobenzaldehyde ; 2-nitro-4′-aminodiphenylamine-4-sulphonic acid with 1-chloro-2-nitrobenzene-4-sulphonic acid ; 4-aminodiphenylamine-2-sulphonic acid with 1-chloro-2 : 6-dinitrobenzene or its 4-sulphonic acid ; 4-aminodiphenylamine with 1-chloro-2 : 6-dinitrobenzene-4-sulphonic acid, 1-chloro-2 : 4 : 6-trinitrobenzene, or 1-chloro-2 : 4-dinitronaphthalene. C. HOLLINS.

Manufacture of acid dyes of the rhodamine series. DURAND & HUGUENIN SOC. ANON. (E.P. 251,644, 29.4.26. Conv., 4.5.25).—Acid rhodamines are obtained in a single operation by condensing a substituted *m*-aminophenol with phthalic anhydride in presence of concentrated sulphuric acid, preferably 100%. The dye from *o*-tolyl-*m*-aminophenol is redder, that from *m*-2-xylyl-*m*-aminophenol yellower, than the corresponding dye obtained by separate sulphonation. The process is applicable also to substituted phthalic anhydrides. C. HOLLINS.

Production of hydroxy-compounds [from primary amines]. J. W. C. CRAWFORD and F. G. WILLSON (E.P. 274,960, 28.4.26).—An arylamine is diazotised, and the diazo compound decomposed at a suitable temperature, in presence or absence of catalysts, the reaction mixture being agitated with an inert solvent which removes the phenol as formed. The solvents used are xylene (for phenol, *p*-cresol, *p*-chlorophenol, *m*-xylenol, salicylic acid), chlorobenzene (for *m*-hydroxybenzaldehyde), and anisole (for phenol). C. HOLLINS.

Catalytic methylation of ammonia and organic amines. A. MACKERT (E.P. 275,377, 17.6.26).—Dimethyl ether is passed with ammonia, or a primary or secondary amine, over alumina, zirconia, thoria, or titanium oxide at suitable temperature and pressure. Aniline with 100% excess of dimethyl ether over alumina at 270—310° gives in two treatments pure dimethylaniline ; with 500—600% excess only one treatment is necessary. β-Naphthylamine, with 600% excess of dimethyl ether over thoria, at 330—350°, gives dimethyl-β-naphthylamine. A mixture of methyl alcohol and dimethyl ether may be used. C. HOLLINS.

Catalytic oxidation of hydrocarbons. E. C. R. MARKS. From E. I. DU PONT DE NEMOURS & CO. (E.P. 275,321, 3.5.26).—In the catalytic oxidation of hydrocarbons (*e.g.*, naphthalene to phthalic anhydride) the gas mixture is preheated by means of preliminary tubes immersed in a bath of molten conducting substance (*e.g.*, a mixture of sodium and potassium nitrates, or a metal

other than mercury) which also surrounds the catalyst tubes and removes excess heat from the reacting mixture.

C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND A.-G., Assees. of P. NAWIASKY, K. ZAHN, and K. SAURWEIN (U.S.P. 1,633,997, 28.6.27. Appl., 23.11.25. Conv., 13.12.24).—See F.P. 607,135; B., 1927, 647.

Preparation of derivatives from vat dyestuffs and alkyl esters of monochloroacetic acid. B. WYLAM, J. E. G. HARRIS, and J. THOMAS, Assrs. to SCOTTISH DYES, LTD. (U.S.P. 1,640,018, 23.8.27. Appl., 19.8.26. Conv., 5.8.25).—See E.P. 263,898; B., 1927, 183.

Manufacture of leuco-hydroxyanthraquinones. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 274,966, 29.4.26).—See G.P. 436,526; B., 1927, 470.

Manufacture of condensation products and dyes of the benzanthrone series [*iso*dibenzanthrones]. I. G. FARBENIND. A.-G., Assees. of A. LÜTTRINGHAUS, H. NERESHEIMER, and H. WOLFF (U.S.P. 1,633,866, 28.6.27. Appl., 16.10.25. Conv., 25.10.24).—See E.P. 255,277; B., 1926, 780.

Preparation of [3-]nitro-[4-]aminobenzoyl-*o*-benzoic acid and derivatives. NEWPORT CO. (E.P. 265,545, 20.10.26. Conv., 8.2.26).—See U.S.P. 1,614,584; B., 1927, 212.

Manufacture of perylene. C. H. MARSCHALK (U.S.P. 1,639,658, 23.8.27. Appl., 13.12.23. Conv., 21.12.22).—See E.P. 208,722; B., 1924, 654.

Reaction product of sulphur chloride and primary arylamines. R. HERZ, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,637,023, 26.7.27. Appl., 31.8.21. Conv., 11.4.14).—See E.P. 17,417 of 1914; B., 1921, 619 A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination and significance of the solubility of artificial silks in alkali. W. WELTZIEN (Papier-Fabr., 1927, **25**, Fest- u. Ausland-heft, 66—71).—Although in the determination of α-cellulose in wood-pulps the material is extracted with 17·5% sodium hydroxide, it is to be remembered that this concentration of alkali is selected on account of its relation to the viscose process, and not because it represents a maximum point of solubility of cellulose. Such maximum solubility exists at a concentration of 10% sodium hydroxide, and important differences in the chemical characteristics of the various types of artificial silk can be detected by exhaustive treatment with sodium hydroxide, preferably at 9% concentration. 0·5 g. of chopped artificial silk is treated in a glass-stoppered flask with 30 c.c. of the sodium hydroxide solution for about 3 hrs. at 18—19°. The liquor is filtered off through a Schott's glass filter-crucible (size of pores 3/5—7), and the residue is pressed hard. The latter is stirred up again with 10 c.c. of fresh soda, allowed to remain for 15 min., and again sucked dry. Afterwards it is washed and dried at 110—115°. The maximum solubility is found in all cases with 10% sodium hydroxide, but 9% affords a more highly-differentiated characterisation of the various types. Cuprammonium silk is the most resistant, with a maximum solubility of 30%; viscose, 40—50%, whilst nitro-silk is almost completely soluble in 10% sodium hydroxide. Finer differentiations between silks of the same type may be made by performing the extraction with sodium hydroxide of different concentrations in the neighbourhood of 10%. The size of the individual filament has an influence on the result, and particular attention must be paid to the temperature.

J. F. BRIGGS.

Digestion of beech wood with nitric acid. H. SUIDA and H. SADLER (Papier-Fabr., 1927, **25**, Fest- u. Ausland-heft, 93—97).—Trials have shown that the nitric acid process is very suitable for the preparation of cellulose from beech wood. This wood has a short fibre and relatively high density, and the usual method of cutting cross-grain chips gives poor penetration and short-fibred pulp. The best results are obtained by slicing the wood longitudinally into slices 1—2 mm. thick. The wood is steeped in nitric acid of the correct concentration at the ordinary temperature for 2—3 hrs. The acid is then drained off and heated up to the desired digestion temperature separately; it is next returned to the digester, which is immersed in a water-bath maintained at 79°. Digestion is complete in about 1 hr.; the acid is drained off, and the chips which retain their shape are washed with water. Disintegration only takes place during the next process, which consists in a treatment with hot 2% sodium hydroxide solution, by which the incrusting matters are dissolved and the cellulose is liberated. A modification of the process, due to Lifschütz, consists in treating the wood with a mixture of sulphuric and nitric acids, but this results in poorer yields and more highly modified cellulose as the concentration of sulphuric acid is increased. It is noted that in the course of the action of the nitric acid on the wood the temperature in the digester rises above that in the surrounding water-bath, due to an exothermic reaction, which must be controlled. The most favourable conditions are obtained with 15% nitric acid and a water-bath temperature of 79°; the time should not be prolonged beyond 90 min. to preserve yield and quality. The consumption of nitric acid is 28—29%. Cellulose of very high quality may be obtained by an after-treatment with 9% sodium hydroxide in the cold.

J. F. BRIGGS.

Action of strong sodium hydroxide on cellulose. P. WAENTIG (Papier-Fabr., 1927, **25**, Fest- u. Ausland-heft, 112—115).—If cellulose is saturated with twice its weight of strong caustic soda solution and stored it undergoes an "ageing" process whereby the viscosity of its solutions is progressively diminished and its solubility in sodium hydroxide solutions (so-called β-cellulose) progressively rises. This change in solubility does not go on indefinitely, but comes to an equilibrium after about 30 days at the ordinary temperature with 17·5% sodium hydroxide solution. Starting with a bleached sulphite pulp showing 82·5% of α-cellulose and 4·3% of β-cellulose, total immersion in 17·5% caustic soda produced only insignificant losses of α-cellulose in 115 days, whereas the same pulp, pressed out and allowed to "age," showed α-cellulose reduced to 57% after 30 days. At the same time the β-cellulose was correspondingly increased so that the sum of the α- and

β-celluloses remained practically constant. The difference between total immersion and "ageing" in the pressed condition can also be determined microscopically, the fibre being but little changed by total immersion, whereas the cellulose which had been "aged" broke down under slight pressure to a mass of fibrillæ. Results of a similar order have also been recorded with cotton cellulose. The suggestion is that the cellulose fibre is not morphologically homogeneous, and the separation into soluble substance and fibrillæ can also be partially effected by mechanical shocks (cf. Cross and Bevan, B., 1919, 7 A). In the case now investigated it would appear that small quantities of atmospheric oxygen play a determining part. J. F. BRIGGS.

Recovery of solvents. BODEWIG.—See I.

Action of hæmatin on fabric. WOODROFFE and GILBERT.—See XV.

PATENTS.

Treatment of fibre. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,632,802, 21.6.27. Appl., 10.8.26).—Crude sulphite fibre is passed through a series of treating tanks in counter-current to a flow of water through a series of containers underneath the tanks, the water being circulated from each container to the corresponding tank. Sodium hydroxide solution is added to an intermediate tank of the series. T. S. WHEELER.

Production of retted bagasse fibre. E. C. LATHROP and T. B. MUNROE, Assrs. to DAHLBERG & Co., INC. (U.S.P. 1,633,594, 28.6.27. Appl., 20.6.24).—Bagasse can be freed from material binding the fibres together by the action of a ferment of the mushroom type, *e.g.*, *Psilocybe*. T. S. WHEELER.

Manufacture of textile material for spinning and other purposes. A. G. BLOXAM. From N. V. OCTROOI MAATSCHAPPIJ "VÉDÉ" (E.P. 274,331, 8.11.26).—Crude fibrous vegetable materials such as straw, grasses, and especially agaves and coconut husks are opened suitable for spinning by successive treatment with a solution of a caustic alkali and a saponifiable substance such as a fatty acid or oil. *E.g.*, coir fibres are boiled for ½—1½ hrs. in a 14·5% solution of caustic soda containing 0·25% of copper sulphate and 0·1% of ammonium chloride (on the weight of raw material) as catalysts, and after removal of loose intercellular substances the residual fibrous material is washed with a weak lye, then dipped into molten stearic acid, and dried preferably at about 35°. A. J. HALL.

Manufacture of textiles. C. DREYFUS, Assr. to CELANESE CORP. of AMERICA (U.S.P. 1,634,613, 5.7.27. Appl., 22.12.21).—Cotton yarn is rendered resistant to water by impregnation with cellulose acetate. T. S. WHEELER.

Manufacture of artificial textile threads. ERSTE BÖHMISCHE KUNSTSEIDEFABR. A.-G. (E.P. 258,582, 14.9.26. Conv., 17.9.25).—Hollow threads are produced by incorporating with any of the usual cellulose solutions, either before or during spinning, substances such as oils, fats, waxes, etc. which do not form bubbles of gas or vapour under the conditions of spinning and finishing, are insoluble in the cellulose solutions, and are in such a fine state of division that the spinning process is unaffected by their presence, these substances being finally removed by suitable solvents. With viscose, substances soluble therein such as soaps from which free fatty acids are produced by the action of the precipitating bath, are also used, the acids being finally dissolved out. B. P. RIDGE.

Manufacture of cellulose derivatives. H. DREYFUS (E.P. 275,286, 26.3.26).—Cellulose esters, especially acetates, of very high viscosity are submitted to a prolonged ripening or secondary treatment in the manner described in U.S.P. 1,217,722 (B., 1917, 451), but without permitting substantial degradation of the cellulose nucleus, until they become soluble in suitable solvents (*e.g.*, acetone–water or other mixtures characteristic of the stage to which the ripening is prolonged). Solutions of much lower viscosity than were obtainable in the earlier stages of ripening are formed, which yield films and threads of increased strength and toughness. B. P. RIDGE.

Reducing the viscosity of nitrocellulose and other cellulose esters. S. DE V. SHIPLEY, Assr. to ATLAS POWDER Co. (U.S.P. 1,633,292, 21.6.27. Appl., 14.3.25).—Nitrocellulose is heated at 100° in water containing a buffer to maintain the suspension at p_H 3·1—8·5. T. S. WHEELER.

Process for reducing the viscosity characteristics of cellulose ethers. P. C. SEEL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,635,013, 5.7.27. Appl., 16.12.25; cf. F.P. 447,974; B., 1913, 420).—A solution of a cellulose ether in a mixture of benzene and alcohol is evaporated, the residue, which should have an acidity exceeding p_H 5, being kept at 65° for several weeks. T. S. WHEELER.

Reducing the viscosity of nitrocellulose solutions. E. C. PITMAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,636,319, 19.7.27. Appl., 11.8.21).—A soluble salt of a lower monobasic aliphatic acid, *e.g.*, sodium acetate (0·01—4%), is added to nitrocellulose solution, which is then kept at 25° for several days. T. S. WHEELER.

Manufacture of cellulose acetate [soluble in acetone]. R. BAYBUTT and E. S. FARROW, JUN., Assrs. to EASTMAN KODAK Co. (U.S.P. 1,635,026, 5.7.27. Appl., 20.7.26).—Cellulose acetate powder soluble in chloroform is treated with 10% nitric acid solution at ordinary temperature for about 20 hrs. T. S. WHEELER.

Production of fine artificial silk filaments. VEREINIGTE GLANZSTOFF-FABR. A.-G. (G.P. 440,227, 26.7.18. Addn. to G.P. 308,427; B., 1922, 807).—The precipitation bath consists of a saturated ammonium sulphate solution containing a little mineral acid. L. A. COLES.

Drying alkali-cellulose. P. C. SEEL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,635,012, 5.7.27. Appl., 24.5.21).—Cellulose saturated with alkali solution for subsequent use in the manufacture of ethers is treated with hot air which has been passed over sodium hydroxide to remove carbon dioxide and dried, first with sulphuric acid and then by refrigeration, being finally heated. T. S. WHEELER.

Cellulose ester composition. G. L. SCHWARTZ, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,633,683, 28.6.27. Appl., 15.6.23; cf. U.S.P. 1,558,299;

B., 1926, 66).—Glacial acetic acid is heated with coconut or similar oil to give mixed glycerides of acetic and higher fatty acids, which are of value as softeners for nitrocellulose. T. S. Wheeler.

Fireproofed [cellulose ester] product. C. E. Burke, Assr. to Du Pont Viscoloid Co. (U.S.P.) 1,633,067, 21.6.27. Appl., 2.1.26).—Magnesium carbonate trihydrate is of value for fireproofing fabrics. T. S. Wheeler.

Manufacture of solid cork articles etc. G. Schellhaas (U.S.P. 1,637,877, 2.8.27. Appl., 11.5.26).—A binding medium mixed with the granular substance (*e.g.*, cork) is dried in the open air, the resulting mass being subjected to a pressure of 16,500 lb./sq. in. and heated to 130° without re-expansion on releasing the heat and pressure. H. Royal-Dawson.

Production of film-like bands from cellulose solutions and similar initial materials. E. Czapek and R. Weingand (U.S.P. 1,641,322, 6.9.27. Appl., 15.1.26. Conv., 15.10.21).—See E.P. 254,946; B., 1926, 738.

Medium for protection of fibres (E.P. 275,374).—See VI.

Artificial wood (U.S.P. 1,634,346).—See IX.

Antimony pentasulphide (U.S.P. 1,633,754).—See XIII.

Rubber-cellulose ester compositions (E.P. 274,968).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing union materials consisting of cotton and [viscose] artificial silk. W. Alterhoff (Textilber., 1927, **8**, 795—796).—In dyeing cotton–viscose silk materials, level shades may be obtained if the grey material be scoured for ½ hr. in a boiling bath containing 1 g. of soda ash and 0·5 g. of Monopole soap per litre, and then entered into a boiling liquor containing 20% of Glauber's salt and 1 g. per litre of a soluble oil (*e.g.*, Monopole Brilliant oil or Avirol KM), the dye being added after ¼ hr. and dyeing continued for ¾—1 hr. at the boil and ½ hr. in the cooling dye bath. The dyed material is then washed and soured in a warm bath containing acetic or formic acid, a small quantity of a suitable oil being added if a soft handle be desired. A. J. Hall.

Dyeing wool and silk with azo dyes produced on the fibre. E. T. Orloff (Textilber., 1927, **8**, 794—795).—Insoluble azo dyes are satisfactorily applied to animal fibres by the following modification of the method used for dyeing cotton. The textile material is impregnated with an acid solution containing the base and a sulphonic acid derivative of the naphthol, and then passed through a 2% solution of sodium nitrite for development of the colour. *E.g.*, 72 g. of β-naphthol are dissolved in 110 g. of sulphuric acid (*d* 1·84) at room temperature, 75 g. of *p*-toluidine added, and the resulting crystalline magma is separated and dissolved in hot water, sodium acetate being then added. Wool or silk is impregnated with the resulting solution, passed through a 2% solution of sodium nitrite, washed with water and also with a solution of an ammonium salt, and dried, the resulting shade being level and fast. A. J. Hall.

Influence of tension and conditions of coagulation on the dyeing properties of viscose silk. A. Lehner and A. Jäger (Kunstseide, 1927, **9**, 219—221, 264—266).—The dyeing and elastic properties of viscose threads are considerably affected by stretching during their coagulation immediately after spinning, and uniform conditions of spinning are essential for the manufacture of viscose silk of a uniform grade. Viscose threads subjected to tension while passing through the usual coagulating bath have less affinity than unstretched threads for direct (Triazole Blue 4B) and vat dyes (Indanthrene Blue BCS), but a greater affinity for basic dyes (Methyl Violet B), this variation of dyeing property being also dependent on the distance from the squirting orifice at which the stretching is applied. After complete conversion of the viscose into cellulose threads, stretching has no appreciable effect. The length of viscose thread immersed in the coagulating bath during the manufacture of viscose silk affects its dyeing properties. A. J. Hall.

Identification of vat dyes on cotton. M. Vais (Textilber., 1927, **8**, 611—619).—Tables are given of the reactions of about 400 vat dyes towards sodium hyposulphite, cold and hot caustic soda (*d* 1·384), concentrated sulphuric acid, concentrated nitric acid, benzol, and pyridine. A. J. Hall.

Patents.

Dyeing cotton with vat and azo dyes. I. G. Farbenind. A.-G., Assees. of Chem. Fabr. Griesheim-Elektron (E.P. 252,384, 20.5.26. Conv., 20.5.25).—Cotton is padded with a coupling component and a leuco-vat dye; the latter is oxidised during coupling with a diazo compound. Leuco-Algol Blue 3G and bisacetoacetyltolidine, developed with diazotised 2 : 5-dichloroaniline or *o*-chloroaniline, give a green; leuco-Algol Blue 3G and 2 : 3-hydroxynaphthoic 2 : 5-dimethoxyanilide, developed with diazotised 2 : 5-dichloroaniline, a brown; leuco-Algol Brilliant Violet R and 2 : 3-hydroxynaphthoic *m*-nitroanilide, developed with diazotised 4-nitro-*o*-anisidine, a blue-red. C. Hollins.

Process of dyeing silk. E. L. Maupai (U.S.P. 1,635,761, 12.7.27. Appl., 1.2.26).—The shades obtained on silk by dyeing with a chromogen, autochrome, or indigosol dye, followed by treatment with a metallic salt, *e.g.*, potassium dichromate, can be rendered fast to boiling soap solution by immediate immersion in a boiling soap solution. T. S. Wheeler.

Dyeing animal fibres. Durand & Huguenin A.-G. (E.P. 255,501, 20.7.26. Conv., 20.7.25).—Certain acid dyes, including the water-soluble ester salts of vat dyes, which do not yield shades on wool fast to rubbing, give satisfactory results in this respect if dyeing is effected in the presence of fuller's earth, due allowance being made for the alkalinity of this assistant. Improved dyeings are thus obtained with Diamond Black F and indigo, if about 20 g. of fuller's earth per litre of dye liquor are used. A. J. Hall.

Dyeing animal fibres [with indigosols]. Durand & Huguenin A.-G. (E.P. 267,986, 22.3.27. Conv.,

22.3.26. Addn. to E.P. 255,501; cf. preceding abstract). —The process of the prior patent is modified by adding the fuller's earth to the oxidising bath instead of (or as well as) to the dyeing bath. C. Hollins.

Method and apparatus for [spot] dyeing [yarn]. Eclipse Textile Devices, Inc., Assees. of H. E. van Ness (E.P. 267,548, 11.3.27. Conv., 11.3.26).—Cross-wound bobbins of yarn are dyed in one or more predetermined parts (spot dyeing) by utilising centrifugal force to cause dye liquor to pass rapidly through limited portions of the yarn. Each bobbin is rotated about a vertical or horizontal axis at 2000 r.p.m., and dye liquor delivered to the centre of the cone is suitably distributed by a spaced perforated cone closely fitting therein, so that the dye liquor is flung outwards through the yarn in layers or zones. Alternatively, internal cones are dispensed with, the distribution of the dye liquor being effected by suitable delivery of the dye liquor. A. J. Hall.

Dyeing cellulose acetate silk. O. Y. Imray. From I. G. Farbenind. A.-G. (E.P. 275,307, 1.5.26).—An aminonaphthoic acid is used as diazo or coupling component for azo dyes for acetate silk either in substance or on the fibre. Examples are: 4-nitro-*o*-anisidine → 2:3-aminonaphthoic acid (red-violet); 2:6-aminonaphthoic acid → 2:3-hydroxynaphthoic acid → β-naphthol (ruby); 2:3-aminonaphthoic acid → β-naphthylamine (brown); 2:6-dimethoxyaniline → 2:6-aminonaphthoic acid (yellow). 33 other couplings are mentioned. 1-Amino-2-ethoxy-6-naphthoic acid is prepared from 2:6-hydroxynaphthoic acid by ethylation, nitration, and reduction. C. Hollins.

Dyeing cellulose esters and ethers. British Dyestuffs Corp., Ltd., R. S. Horsfall, L. G. Lawrie, and J. Hill (E.P. 275,373, 11.6.26).—Acetate silk etc. is dyed with azo dyes prepared by coupling a diazotised arylaminesulphonarylamide with an arylamine or an unsulphonated derivative thereof. *m*-Aminobenzenesulphonanilide → α-naphthylamine gives an orange; *m*-aminobenzenesulphon-*m*-nitroanilide → cresidine, an orange-red; α-naphthylamine-5-sulphonanilide → cresidine, a deep yellow. The arylamides are obtained from nitro- or acylamino-arylsulphonyl chlorides and arylamines, with subsequent reduction or hydrolysis. C. Hollins.

Dyeing viscose filaments and films. W. Mendel, Assr. to S. A. Neidich (U.S.P. 1,633,220—1, 21.6.27. Appl., 23.5.25).—(A) Coagulated viscose is heated in 3% aqueous sodium phosphate solution at 75° for 2 hrs., washed, and dyed in any usual manner. (B) The dye is added to the sodium phosphate solution. T. S. Wheeler.

Manufacture of a medium for the protection of fibrous materials during the chemical treatment thereof. V. Haberkorn (E.P. 275,374, 11.6.26).—A solution of wool waste in aqueous sodium hydroxide is boiled and then made slightly acid. A protective medium is obtained, in solution or in solid form according to the proportions used, which when added to an acid dye-bath ensures level dyeing and prevents attack of the fibre. Dyeing and chroming are also accelerated. C. Hollins.

Treatment of fibrous materials and textiles. [Use of wetting-out agents in oiling, sizing, etc.] I. G. Farbenind. A.-G. (E.P. 246,468, 18.1.26. Conv., 21.1.25).—Aromatic sulphonic acids (or their salts) which have strong wetting-out action, particularly propyl- and butyl-naphthalenesulphonic acids, benzylsulphanilic acid, sulphonated formaldehyde-phenol or formaldehyde-naphthalene condensation products, etc., are added to the liquors used in the oiling, sizing, or similar treatment of textiles etc. C. Hollins.

Ornamentation of artificial silk fabrics. Calico Printers' Assoc., Ltd., and J. R. Whinfield (E.P. 275,357, 25.5.26).—Acetate silk is printed with suitably thickened delustring salts (aniline hydrochloride, sodium phenoxide, etc.) and then treated with dilute alkali or acid to decompose the salt, a matt pattern on a lustrous ground being produced. By using slight excess of alkali with an acid delustring agent (phenol) partial hydrolysis results and the matt pattern may be dyed with direct dyes. For lustrous patterns on a matt ground the fabric may be first printed with a suitable resist, *e.g.*, a paste of zinc oxide, and then padded with the delustring agent; dye may be added to the resist paste, or free alkali may be used and the lustrous, partially hydrolysed pattern may be subsequently dyed. C. Hollins.

Mercerising or otherwise treating with caustic alkalis textile materials containing viscose artificial silk. Silver Springs Bleaching & Dyeing Co., Ltd., F. E. Mason, and A. J. Hall (E.P. 274,266, 2.7.26). —Cotton materials containing viscose silk which are to be mercerised are manufactured with non-desulphurised silk, since this is more resistant than desulphurised silk to caustic soda. After mercerisation the material is subjected to the usual processes for desulphurising viscose silk. A. J. Hall.

Treatment of cotton or other threads containing cellulose. G. Tagliani, Assr. to Chemical Works (formerly Sandoz) (U.S.P. 1,633,617, 28.6.27. Appl., 29.6.25. Conv., 3.7.24: cf. E.P. 233,704; B., 1925, 956).—Cotton which has been substantively dyed is rendered indifferent to the further action of such dyes by treatment first with alcoholic sodium hydroxide solution and then with *p*-toluenesulphonyl chloride dissolved in carbon tetrachloride. T. S. Wheeler.

Manufacture of finished [regenerated cellulose] fabric. J. Bennett, Assr. to Consolidated Textile Corp. (U.S.P. 1,633,160, 21.6.27. Appl., 26.8.26).—The lustre of regenerated cellulose fibres can be removed by treatment with sodium hydroxide solution, patterns being obtained by using a resist paste. T. S. Wheeler.

Process of finishing fabrics [viscose]. W. Witte, Assr. to U.S. Finishing Co. (U.S.P. 1,633,152, 21.6.27. Appl., 29.6.26).—Viscose silk is delustred by treatment with alcoholic sodium hydroxide solution, followed by immersion in a solution of *p*-toluenesulphonyl chloride in carbon tetrachloride. The process can be adapted to printing purposes. T. S. Wheeler.

Dyeing. I. G. Farbenind. A.-G., Assees. of W. Herzberg and H. Ohlendorf (U.S.P. 1,640,657, 30.8.27. Appl., 4.10.26. Conv., 9.6.25).—See E.P. 253,488; B., 1927, 551.

Anthraquinone dyes and dyeing (E.P. 275,421).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Alkali chloride industry. J. Billiter (Z. Elektrochem., 1927, **33**, 353—360).—A lecture.

Steels for ammonia synthesis. Vanick.—See X.

Electrolysis of water. Sestini.—See XI.

Ammonium sulphate and soil acidity. Hagen.—See XVI.

Determination of sulphuric acid by means of benzidine. Haase.—See XXIII.

Patents.

Production of sulphurous acid for the manufacture of sulphite solutions. A.-G. für Zellstoff- u. Papierfabr. (G.P. 440,337, 22.9.25).—Finely-ground pyrites or the like is injected into a combustion chamber wherein it is burnt in a regulated supply of primary or secondary air. L. A. Coles.

Production of hydrated lime. W. K. Hunter, Assr. to M. F. Nichols (U.S.P. 1,634,424, 5.7.27. Appl., 22.7.25).—Lime is discharged from the kiln without cooling into an excess of water. T. S. Wheeler.

Production of solid ammonium carbonate from its components. Rhenania-Kunheim Ver. Chem. Fabr. A.-G. (E.P. 275,459, 20.11.26. Addn. to E.P. 262,408; B., 1927, 652).—Ammonium carbonate produced from gaseous ammonia and carbon dioxide is improved in quality and hardness if the temperature within the lead or aluminium reaction vessel is kept below 60°, preferably 35—45°, either by adjusting the amounts of the reagents so that the heat of formation maintains the desired temperature or by artificial cooling or external heat. The gases may be introduced under pressure, in which case the temperature is maintained at 60—90°. W. G. Carey.

Purification of alkali cyanide solutions containing sulphides. I. G. Farbenind. A.-G., Assees. of K. Herrdegen (U.S.P. 1,633,485, 21.6.27. Appl., 27.11.26. Conv., 4.12.25).—Crude cyanide solutions, obtained in the Bucher process, can be freed from sulphides by treatment with bismuth hydroxide. T. S. Wheeler.

Production of potassium nitrate. Preussische Bergwerks- u. Hütten-A.-G., Abtg. Salz- u. Braunkohlenwerke (F.P. 616,286, 17.5.26. Conv., 16.6.25).—Double decomposition of a crude potassium salt, *e.g.*, the chloride, and sodium nitrate is effected in the presence of saturated potassium nitrate solution. L. A. Coles.

Production of barium oxide. Rhenania-Kunheim Ver. Chem. Fabr. A.-G. (G.P. 440,382, 1.5.23).—A mixture of barium carbonate and carbon packed in containers constructed of paper etc. is heated strongly. L. A. Coles.

Manufacture of precipitated barium carbonate and barium sulphydrate. J. B. Pierce, jun. (U.S.P. 1,634,338, 5.7.27. Appl., 18.11.24).—Barium sulphide solution is treated with pure carbon dioxide at 80° until one half of the barium present has been precipitated as barium carbonate. The resulting barium hydrogen sulphide solution is concentrated. T. S. Wheeler.

Process of refining barytes. W. J. O'Brien (U.S.P. 1,633,347, 21.6.27. Appl., 17.9.25).—Barytes ore is extracted with fuming sulphuric acid, dissolved barium sulphate being precipitated by volatilising sulphur trioxide from the solution. T. S. Wheeler.

[Calcining] treatment of [finely-divided] carbonate materials. A. E. White. From Dwight and Lloyd Metallurgical Co. (E.P. 275,463, 26.11.26).—Limestone, dolomite, etc., when in small particles, is calcined by spreading a graded layer, with the larger particles in the upper portion, upon a pervious support and passing highly heated gases through the layer from the top downwards until the carbonate is dissociated, maintaining an excess of heated gases above the layer and removing the gases from below, together with the carbon dioxide as it is formed. The material is separated from the pervious support by a thin layer of previously treated similar material, and may be heated more quickly by admixing solid fuel insufficient for complete calcination. The process may be carried on while the material is being transported bodily through a treatment zone. W. G. Carey.

Production of sulphites. Bradley-McKeefe Corp., Assees. of L. Bradley and E. P. McKeefe (Can. P. 263,180, 25.8.24).—Dolomitic limestone or lime is converted into calcium and magnesium bisulphites by treatment with sulphur dioxide, and the product is treated with sodium sulphate in the presence of excess sulphur dioxide. L. A. Coles.

Manufacture of hypochlorites. R. B. MacMullin, Assr. to Mathieson Alkali Works (U.S.P. 1,632,483—5, 14.6.27. Appl., [a, b], 22.8.25; [c], 23.9.25; cf. E.P. 195,366; B., 1924, 173).—(a) Aqueous hypochlorous acid is treated with a tertiary alcohol, *e.g.*, *tert.*-amyl alcohol, the corresponding alkyl hypochlorite which separates being dissolved in a liquid in which water is also soluble, *e.g.*, amyl alcohol, and treated with a limited amount of water and calcium oxide, to form solid calcium hypochlorite with regeneration of the alcohol. The alkyl hypochlorite can also be formed directly by passing chlorine into a solution of sodium hydrogen carbonate in presence of the alcohol. (b) A solution of pure calcium hypochlorite is treated with excess of calcium oxide to precipitate basic calcium hypochlorite. (c) A solution of pure calcium hypochlorite can be obtained by treating a *tert.*-alkyl hypochlorite prepared as in (a) with water and a limited quantity of calcium oxide. T. S. Wheeler.

Production of hyposulphites by the action of amalgams on bisulphite solutions. I. G. Farbenind. A.-G., Assees. of F. Bencker (G.P. 440,043, 4.3.21).—Bisulphite solutions are treated with an amalgam and a quantity of free acid equivalent to the amalgam. L. A. Coles.

Production of solid sodium bisulphite or sulphite. Chem. Fabr. Kalk G.m.b.H., and H. Oehme (G.P. 440,380, 17.2.25).—Pure sodium bicarbonate, formed by treating sodium carbonate with carbon dioxide liberated

during the process, is treated with sulphur dioxide in the presence of saturated sodium bisulphite solution.
L. A. COLES.

Production of anhydrous sodium bisulphite. N. A. LAURY (U.S.P. 1,638,492, 9.8.27. Appl., 16.8.23). —Crude sodium carbonate is subjected to the action of sulphur dioxide in the presence of a trace of moisture.
H. ROYAL-DAWSON.

Treatment of aluminous ores. R. MOLDENKE (U.S.P. 1,637,451, 2.8.27. Appl., 9.7.25).—Part of the aluminium contained in the ore (alunite) is converted into potash alum, which is separated, and the remainder into an ammonium aluminium alum, both products being then treated with ammonia solution to form aluminium hydroxide. H. ROYAL-DAWSON.

Production of aluminium sulphate. GEN. CHEMICAL Co., Assees. of R. M. MEIKLEJOHN (Can. P. 263,596, 2.10.25).—A mixture of aluminous material with a quantity of sulphuric acid greater than 1/1·3 of the water content of the mixture is heated above 160°.
L. A. COLES.

Production of aluminium fluoride. E. TEISLER (Can. P. 263,352, 20.5.25).—Finely-divided clay or other material containing silica in addition to alumina is treated with hydrofluoric acid, and the aluminium fluosilicate formed is converted into aluminium fluoride by treatment with material containing aluminium oxide or hydroxide. L. A. COLES.

Preparation of magnesium chromates. G. KRÄNZLEIN and A. VOSS, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,632,299, 14.6.27. Appl., 3.6.26. Conv., 22.6.25).—Magnesium chromate can be readily prepared by precipitating magnesium sulphate solution with calcium chromate at the b.p. for 6—8 hrs., collecting the precipitated calcium sulphate, and evaporating the filtrate to separate the magnesium salt. Magnesium dichromate, owing to its great solubility, can be obtained from the interaction of magnesium chloride and sodium dichromate solutions in the cold, the requisite evaporation being carried out *in vacuo*. T. S. WHEELER.

Production of phosphorus nitride and volatile halides from raw materials. C. G. MINER (U.S.P. 1,634,795—6, 5.7.27. Appl., 25.7.25).—Phosphate rock is treated with sodium chloride at 1300° to give phosphorus pentachloride, which is passed at 500° over aluminium nitride, prepared by heating a mixture of bauxite and carbon at 1500° in an atmosphere containing a hydrocarbon and nitrogen. Phosphorus nitride, of value as a fertiliser, and aluminium chloride are obtained. Silicon nitride reacts similarly to give silicon tetrachloride.
T. S. WHEELER.

Separation of nitrosyl chloride from chlorine. W. DOMINIK (G.P. 440,334, 23.8.25. Conv., 22.12.24).—A mixture of nitrosyl chloride with chlorine is brought in contact with nitric acid of *d* not less than 1·332, at 20—30°, whereby the nitrosyl chloride liquefies without the chlorine entering into solution. L. A. COLES.

Production of ammonium chloride and alkali sulphate. CONTINENTALE A.-G. FÜR CHEMIE, Assees. of O. GERNGROSS (U.S.P. 1,634,870, 5.7.27. Appl., 22.5.25. Conv., 11.6.24).—See E.P. 235,552 ; B., 1925, 714.

Manufacture of alkali sulphides. H. FREEMAN, Assr. to CANADA CARBIDE Co. (U.S.P. 1,640,314—5, 23.8.27. Appl. [A], 14.2.23, [B], 30.6.24).—See E.P. 211,488 ; B., 1925, 630.

Manufacture of pure barium carbonate. F. FALCO (U.S.P. 1,640,652, 30.8.27. Appl., 8.9.26. Conv., 27.11.25).—See E.P. 270,559 ; B., 1927, 522.

Manufacture of lead monoxide by a wet method. J. J. TARDAN (E.P. 265,190, 21.1.27. Conv., 30.1.26).—See F.P. 611,810 ; B., 1927, 389.

Recovery of oxides of nitrogen in sulphuric acid manufacture. A. M. FAIRLIE (Re-issue 16,709, 23.8.27, of U.S.P. 1,420,477, 20.6.22).—See B., 1922, 630 A.

Pulverising machine (U.S.P. 1,633,164).—See I.

Recovery of copper, zinc, and other metals from solutions containing chlorides and sulphates (E.P. 276,200).—See X.

Electrolysis of water (E.P. 275,785 and G.P. 440,335).—See XI.

Lead compounds of chromic acids (U.S.P. 1,633,948). **Azides** (G.P. 440,568).—See XIII.

VIII.—GLASS ; CERAMICS.

Ancient glass. II. B. NEUMANN [with M. RUPPRECHT] (Z. angew. Chem., 1927, **40**, 963—967 ; cf. Neumann and Kotyga, B., 1925, 992).—In continuation of earlier work on ancient glasses analyses are given of two Egyptian samples from Thebes (B.C. 1500), eight mosaic cubes from Salona (200 A.D.), and five glasses from Samarra (850 A.D.). The Egyptian glasses contained 67—62% of silica, 16—20% of alkali oxides, 4—5% of lime, 2—4% of magnesia, 4—1·5% of alumina, and 1—2% of ferrous oxide, and were coloured dark blue with cupric and manganous oxides ; it is suggested that these "dolomitic" glasses high in alumina are characteristic of Egyptian work. The Roman glass cubes resembled in composition those already described in the earlier work, but the Mesopotamian samples were of an entirely distinct type, being characterised by a high magnesia and a relatively low alkali content. The high degree of transparency of these glasses combined with the high "dolomite" content (9—11% CaO + MgO) shows that a high temperature must have been used in making them. The Egyptian and Mesopotamian glasses have a softening point about 100° higher than that of the Roman and Rhenish ancient glasses. A. R. POWELL.

Whiteware bodies developed at the Ohio State University. A. S. WATTS (J. Amer. Ceram. Soc., 1927, **10**, 148—149).—The compositions of bodies suitable for statuary, electrical, hotel, and chemical porcelain and compounded mainly from American raw materials, together with glaze formulæ, are given. In bodies containing a maximum of 15% of ball clay, 50% of total clay, and 24% of felspar, the best colour is produced by the addition of not more than 2·25% of calcium and magnesium carbonates in the proportion of 60% $MgCO_3$ and 40% $CaCO_3$. F. SALT.

Sheet steel cooking ware white enamels economically suitable for use in China. CHI-FANG LAI

(J. Amer. Ceram. Soc., 1927, 10, 150—162).—Two typical ground coats, one high in felspar and cobalt oxide, the other high in quartz and cryolite, and four cover coats were studied. A number of opacifiers were substituted systematically for tin oxide. Test pieces were fired in a simple oil-fired muffle furnace. The effect of varying the composition was more marked in the case of the cover coats than with the ground coats. The cheapest enamels, resembling in quality those made with tin oxide, were obtained with sodium antimonate and calcined alumina. Zirconium oxide increased the resistance to thermal shock and to acids, whilst sodium antimonate increased the impact strength. The best cover coat from the point of view of economy had the refractory portion of base enamel high in felspar, low in quartz, and contained a medium amount of cryolite. Consistently good results were obtained with a coat in which felspar was the only refractory and which contained no cryolite. The gloss was considerably improved, but the opacity impaired, by a high zinc oxide content. F. Salt.

Effects of composition on the properties of ground-coat enamels for sheet steel. W. N. Harrison and H. G. Wolfram (J. Amer. Ceram. Soc., 1927, 10, 163—179).—Three series of ground coats, 20 in each series, were prepared on three refractory bases, one consisting of 33·5 pts. of felspar and 25 pts. of flint, another of felspar only, and a third of flint only. A fixed addition containing 0·5 pt. of cobalt oxide, 1 pt. of manganese oxide, and 5 pts. each of sodium oxide and boric oxide was also made to each of the enamels. In each case the refractory base and the fixed addition were present in the proportion of 58·5% and 11·5%, respectively, making a total constant portion of 70%. The remaining variable portion (30%) was made up of equal parts of three constituents taken from six fluxes commonly used in enamels. Two standard white cover coats were applied to each of the ground coats, and the enamels were tested for resistance to mechanical and thermal shock. A high felspar content in ground coats produced greatly decreased resistance to mechanical and thermal shock. On the other hand, a high flint content slightly impaired the resistance to thermal shock but greatly improved the resistance to mechanical shock. In the variable portion of the ground coats, sodium oxide produced the most favourable results under the tests. A high boric oxide content was also effective in ground coats containing both felspar and flint. The effect of expansivity on resistance to shock was obscured by other factors. F. Salt.

Variations in softening points of refractory materials due to rate of heating. S. S. Cole (J. Amer. Ceram. Soc., 1927, 10, 180—184).—Softening point tests were carried out on a number of refractory materials, including fire clay, silica, and silica cements, in a gas-fired furnace. The rate of heating up to cone 28 was varied from 1 to 5 hrs. Long heating did not appreciably lower the softening point of fire clays. With silica bricks the 1½ hr. rate gave the highest results; longer heating reduced the softening point. Results with silica cements were erratic. Variations in softening temperature were reduced by fine grinding and intimate mixing of the materials; the purer the material the less the softening point varied. F. Salt.

Patents.

Manufacture of glass containing barium. Rhenania-Kunheim Ver. Chem. Fabr. A.-G. (E.P. 259,953, 8.10.26. Conv., 16.10.25).—Barium silicate is employed, in conjunction with the usual batch materials (sand, soda ash, lime-spar), in preparing glasses containing barium. The silicate is obtained from heavy spar by the method described in E.P. 242,996 (cf. B., 1926, 787). A. Cousen.

Production of opaque, readily-fusible glass or enamel. Patent-Treuhand-Ges. f. elektr. Glühlampen m.b.H., Assees. of F. Skaupy, F. Späte, and H. Nachod (G.P. 440,298, 5.12.24).—Insoluble compounds of boron or silicon with other metalloids or with acids, such as boron nitride or phosphate, or the corresponding silicon compounds, are added to the glass flux. L. A. Coles.

Manufacture of an abrasive article. L. H. Milligan and C. H. Quick, Assrs. to Norton Co. (U.S.P. 1,635,981, 12.7.27. Appl., 4.9.25).—Granulated alumina is mixed with fusible clay and shaped, the article being then coated with a mixture of ground felspar (82%), plastic ball clay (15%), and lime (3%), and fired. T. S. Wheeler.

Production of silica articles. British Thomson-Houston Co., Ltd., Assees. of H. L. Watson (E.P. 268,780, 29.3.27. Conv., 1.4.26. Cf. E.P. 230,499; B., 1925, 850).—Silica is heated to plasticity by means of imbedded electric resistances around a core of refractory material. The plastic silica is withdrawn through a tubular die of refractory material, the rate of withdrawal determining the nature of the product. Unfused silica is excluded from the die, and the product is protected from the heat radiated from the fusion zone. B. W. Clarke.

Luminous enamels. P. F. Jooss and H. E. Ficheroulle (F.P. 610,454, 9.5.25).—The metal surface to be enamelled is coated with a deoxodising layer, then with a mixture of lead-free flux and unground phosphorescent zinc sulphide, with or without a small quantity of radioactive substance. The article is then fired rapidly until the flux just melts. The process is applicable to the enamelling of glass and ceramic products. A. R. Powell.

Enamelling sheet iron. C. Totot-Gibaru (F.P. 610,645, 5.2.26).—The cleaned sheets are heated at 420° and covered with an enamel containing fluorine, boric acid, zinc oxide, and antimonious oxide in a lead-free flux containing silica, alumina, lime, and alkali. Subsequently a second layer of enamel similar to the first is applied, but containing stannic oxide and pigments such as ferric, manganous, or cobalt oxide in place of the antimony oxide of the first layer. For sanitary ware a second layer similar to the first may be used instead of the coloured layer. A. R. Powell.

Eye-protective glass. W. W. Coblentz, Assr. to Sec. Commerce of United States (U.S.P. 1,637,439, 2.8.27. Appl., 10.6.26).—The glass contains the usual constituents and sufficient cobalt oxide to give a blue colour equivalent to blue 20 + 8 on the Lovibond scale and sufficient cerium oxide to stop ultra-violet rays. H. Royal-Dawson.

Paving bricks etc. (E.P. 275,104). **Enamelling cement** (F.P. 610,956).—See IX.

IX.—BUILDING MATERIALS.

Fire-resistant construction. R. E. Stradling and F. L. Brady (Dept. Sci. Ind. Res., Building Res., Spec. Rep. No. 8, 1927. 57 pp.).—Metals do not resist the effects of fire owing to the loss in strength which occurs at high temperatures. Timber becomes a good fire resister, provided the wood is of large enough dimensions and free access of air is prevented. Natural crystalline rocks, such as granites, as well as limestones, break down suddenly and completely at high temperatures. Sandstone does not lose so much strength, but is very liable to crack owing to shrinkage. Burnt clay products including terra-cotta are in general good fire resisters, although failure may occur if quartz or silica is present. Lime is likely to cause disintegration in any structure owing to the dehydration of the calcium hydroxide, followed by rehydration and expansion on exposure to air and water. Lime-clinker mortars are more resistant than lime-sand mortars, owing to part of the lime being chemically combined. Similarly, concrete is liable to fail owing to its free lime content. Concrete reinforced near the surface is liable to fail owing to the expansion of the metal with subsequent spalling of the concrete. For a fire-resistant concrete an aggregate which will enter into chemical combination with the free lime is necessary. Pozzolanic materials, *e.g.*, granulated slag, spent shale, baked clay, etc., are best for this purpose, with a fine aggregate composed of red brick or dolerite. Portland blast-furnace cement is very suitable for fire-resistant construction. Concrete of this type has the advantage of increasing in strength upon exposure to water after exposure to fire. B. W. Clarke.

Analysis of cements. G. Gallo (Giorn. Chim. Ind. Appl., 1927, **9**, 313—317).—From analyses of cements of known composition before and after setting it is concluded that Kriege's method (cf. B., 1924, 598) for analysis of cements is inaccurate; an alternative method which is thought to be more satisfactory is described. R. W. Lunt.

Distinguishing green from old wood. G. Fron (Ann. Falsif., 1927, **20**, 386—391).—10 g. of the dried wood, cut into strips 2—3 mm. thick, are boiled with 80 c.c. of water for 10 min. and the hydrogen-ion concentration of the cooled extract is determined colorimetrically with Methyl Red. The method is not suitable for woods such as oak or chestnut rich in tannin, owing to the deep colour of the extract. Green woods and those which have been artificially dried by heat show p_H values between 5·2 and 6·5 according to their nature. The naturally dried woods have higher acidities, the p_H varying between 4·4 and 5·2, and similar values are shown by woods artificially aged by ozonised air. F. R. Ennos.

Pigments for mortar. Foster.—See XIII.

Patents.

Manufacture of paving bricks and other ceramic products. Barton Foreign Patents Ltd. From G. D. Cotton (E.P. 275,104, 19.2.27).—The bricks are fired in a continuous kiln which is arranged so that, after passing through a vitrifying zone, the bricks are subjected to a primary devitrifying treatment and then heated again in an annealing zone before reaching the final cooling chamber. The bricks are rendered less brittle, and development of cracks is prevented. B. W. Clarke.

Manufacture of artificial marble. G. Bia and J. E. D. de G. de Bielize (E.P. 275,448, 26.10.26).—A solution containing zinc and magnesium sulphates in definite proportions is treated in a colloid mill with vaseline oil and phenol-formaldehyde condensation products. The viscous colloidal product is made slightly acid with sulphuric acid and added to a mixture of magnesia with water and a suitable filler, such as barium sulphate. B. W. Clarke.

Manufacture of bituminous concrete. L. S. van Westrum (E.P. 275,364, 31.5.26. Addn. to E.P. 269,975; B., 1927, 484).—A cementitious powder, consisting of a mixture of a true cement with stone or slag flour, with or without the addition of oxide of iron, is mixed with a mineral aggregate and with a bituminous soap prepared by saponifying a mixture of bituminous material and a vegetable oil. B. W. Clarke.

Asphaltic paving composition. C. N. Forrest, Assr. to Barber Asphalt Co. (U.S.P. 1,634,241, 28.6.27. Appl., 22.4.25).—Sodium acetate (1—5%) toughens and stabilises asphaltic paving compositions. T. S. Wheeler.

Enamelling articles made of fibrous cement. Soc. Co-op. Emo (F.P. 610,956, 4.1.26. Conv., 19.12.25).—The well-dried article from which all grease has been removed is covered with a thick paste made by mixing stannic oxide, cryolite, and a pigment with powdered flux and water, and, after drying, the article is heated until the flux melts. The flux is made by melting together silica, red lead, borax, and nitre, with subsequent grinding to an impalpable powder. A. R. Powell.

Manufacture of artificial wood. T. Surdi (U.S.P. 1,634,346, 5.7.27. Appl., 12.10.25. Conv., 20.12.24).—Vegetable fibres are dried and fireproofed, being then mixed with a binder, and compressed and heated in moulds. T. S. Wheeler.

Preservation of wood. E. Plank (G.P. 439,523, 6.11.25).—The wood is placed in a solution of lead nitrate (10%) contained in a pressure vessel fitted with an insulating lining having apertures for electrodes of lead and arsenic. The pressure is increased up to 10 atm. and the solution heated electrically to the b.p. by means of an alternating current. After some time the pressure is released, a protective colloid being obtained from the wood. An induction coil is then inserted into the circuit, and a colloidal solution of lead and arsenic formed in the bath by electrical dispersion. The pressure is finally again increased to 10 atm. and the temperature raised to 100°, a colloidal layer being so produced on the outer surface of the wood and on the walls of the capillaries. This layer, which is very difficult to remove by washing, forms an active protection against destructive agents. The process at the same time artificially ages the wood. A. B. Manning.

Material for the manufacture of artificial stone and similar products. R. Schoenhoefer (U.S.P. 1,633,132, 21.6.27. Appl., 28.4.24. Conv., 30.9.20).—See E.P. 235,257; B., 1925, 633.

Composition for roofing, road surfacing and other purposes. L. C. Lutyens and R. O. Child (U.S.P. 1,641,637, 6.9.27. Appl., 6.4.25. Conv., 11.4.24).—See E.P. 238,586; B., 1925, 851.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

High-frequency induction melting. D. F. Campbell (Iron and Steel Inst., Sept., 1927. Advance copy. 9 pp.).—One special advantage in melting metal under high-frequency conditions is the absence of a complicated form of bath necessary in low-frequency melting. Metal may be melted in a crucible supported externally by heat-resisting sand, and, as the heating is not conducted through the crucible walls, maintenance cost of refractory materials is not great. The Ajax-Northrup furnace is described (cf. B., 1927, 683), the method being of particular interest in preparing small quantities of alloy steels, under crucible conditions, for the casting of small parts, *e.g.*, motor-car parts. High-frequency generators of 600 kv.a. are now in regular operation under economic commercial conditions. C. A. King.

Effect of varying ash in the coke on blast-furnace working. C. S. Gill (Iron and Steel Inst., Sept., 1927. Advance copy. 7 pp.).—Fluctuations in the quality of iron from cast to cast were found to follow closely the varying ash content in coke as supplied to the furnace. Later, when working with coke of regular ash content over a period of five weeks, the make increased by 15·4% accompanied by a reduction in coke consumption of 1·01 cwt. per ton of pig, when using coke containing an average ash content of 9·76% as compared with coke containing 12·15%. The practical working of the furnace also improved considerably. C. A. King.

Value of an addition of coal dust in the melting of iron in a cupola. P. Bardenheuer and A. Kaiser (Stahl u. Eisen, 1927, **47**, 1389—1395).—Considerable economies may be effected in iron foundries by supplying about 10—12% of the fuel required in the cupola in the form of coal dust, which is blown into the furnace through a series of small tuyères situated a short distance above the ordinary air-blast tuyères. Comparative tests on the same furnace with the same iron showed that instead of the ordinary coke consumption of 11% of the charge, only 7% was needed if coal dust equal to 0·7—0·8% of the charge was blown in with compressed air The melting zone was thus shortened and the preheating zone lengthened with a corresponding decrease in the amount of carbon monoxide in the flue gases without lowering the temperature of the iron leaving the furnace. At the same time the capacity of the furnace per day was increased by 23—30%, the economic utilisation of the fuel by 37%, and the fuel consumption by 30%, the amount of air blast required being reduced by 20% and the sulphur content of the iron decreased. Owing to the smaller fuel consumption and the smaller amount of ash to be slagged, the coke and limestone consumption were decreased considerably and less slag was produced. Rods cast 30 mm. thick from a furnace fired with coal dust had a bending strength of 46—50 kg./mm.^{2} and a tensile strength of 28—35 kg./mm.^{2} A. R. Powell.

Direct production of pure iron. P. Longmuir (Amer. Electrochem. Soc., April, 1927, **51**. Advance copy. 10 pp.).—A review of the various methods that have been suggested for the production of iron sponge by the low-temperature reduction of iron ore with solid or gaseous reducing agents. The author considers that the ideal method should consist of the following steps: distillation of coal at a low temperature to give a highly reactive coke, gas, and by-products; reduction of the ore, possibly in two stages, by means of producer gas; magnetic separation of the sponge followed by briquetting and melting in electric furnaces; and, finally, regeneration of carbon monoxide from the waste gases by passing them directly from the reduction furnace through a red hot mass of low-temperature coke produced in the first stage of the process. A. R. Powell.

Manufacture of steel in "one process" direct from ore. O. Smalley and F. Hodson (Amer. Electrochem. Soc., April, 1927, **51**. Advance copy. 16 pp.).—In the Pehrson–Prentice process the ore separated from the gangue by means of an electromagnet is mixed with carbon and the mixture charged into a rotating furnace fitted with louvres for the admission of hot gases. Reduction is effected at 950—1000° with a thermal efficiency of 97%. The hot sponge is melted electrically in a gas-tight tilting furnace capable of accurate temperature control to 1800°, and the molten iron is refined and worked into steel as usual. The metal produced compares favourably in mechanical properties with the best crucible steel. Details are given of the mechanical properties of steels produced from New Zealand and Java titaniferous iron sands by the "Carsil" process involving direct reduction of the ore followed by melting and refining in a continuously-operated electric furnace. A. R. Powell.

Mechanism of the tempering of steels. T. Matsushita and K. Nagasawa (Iron and Steel Inst., Sept., 1927. Advance copy. 12 pp.).—Decomposition of martensite during the tempering of steel was followed by measuring the change in electrical resistance and the intensity of magnetisation during the heating. It was indicated that there are two martensites, α decomposing mainly in the interval 100—170° and β decomposing at 170—300°. Complete decomposition of martensite in one specimen was effected by heating at 250° for 2 hrs., and on reheating the specimen in a dilatometer contraction was observed at 300—400°, which leads to the view that the decomposition product of martensite is free carbon, and not cementite, which combines with iron in the range 300—400° to form cementite. Boundary temperatures are proposed for troostite (300°), sorbite (400°), and granular pearlite (550° to Ac1 point). C. A. King.

Non-rusting steels. B. Strauss (Z. Elektrochem., 1927, **33**, 317—321).—The history of the development of non-rusting steels and their properties and applications are briefly reviewed. Further experiments (cf. B.,

1925, 722) on the electrode potential of chromium steels and chromium-nickel steels in ferrous sulphate solutions are described, measurements being made in the presence of hydrogen, air, or hydrogen peroxide. With steels containing 18—20% Cr and 8% Ni, the metal is passive in the presence of air, unless the carbon content exceeds about 1·2%; in the presence of hydrogen peroxide, even with 4% C, a noble potential was obtained. The effect of the annealing temperature on the magnetic properties of chromium-nickel steels is also dealt with.

H. J. T. ELLINGHAM.

Deterioration of alloy steels in ammonia synthesis. J. S. VANICK (Chem. Met. Eng., 1927, 34, 489—492).—Test specimens in the form of cylinders and containing similar specimens for tensile tests etc. were exposed to a temperature of 500°, a pressure of 100 atm., and an ammonia concentration of 8·3% in a nitrogen-hydrogen mixture for several months. The steels used were first annealed. Little change could be observed in 2 weeks. With longer exposures, fissuring and decarbonisation occurred, the penetration being either uniform or of the "tendril" type. Low-carbon steel deteriorated less rapidly than high-carbon steel, though both were penetrated to the core in 4 months, as were also nickel steel and low-chromium steel. A tungsten-chromium steel suffered much less. Further tests showed that a suitable steel would contain 2·25% Cr and not more than 0·3% C, the high chromium content reducing the action to a uniform penetration. High-chromium steels of the stainless type are most resistant, but are too difficult to work. Vanadium offered no advantage. The protective action of chromium results from the formation of a solid solution of chromium nitride. C. IRWIN.

Influence of nickel and silicon on an iron-carbon alloy. A. B. EVEREST, T. H. TURNER, and D. HANSON (Iron and Steel Inst., Sept., 1927. Advance copy. 29 pp.).—The influence of nickel and silicon on a simple iron-carbon alloy (American washed iron) was investigated over a range 0—40% Ni, 0—3·6% Si. The original cast alloy was white and unsound, and small additions of nickel rendered this iron first sound, then grey, though four times as much nickel as silicon was required to produce a given degree of graphitisation, which, however, was finer when induced by nickel. Alloys containing 5—18% Ni were hard and martensitic, becoming soft and non-magnetic with austenitic structure when more than 18% Ni was present, and regaining magnetic properties beyond 20% Ni. The presence of increasing quantity of silicon appeared to mask the effect of nickel. The densities of the alloys, whilst high for grey irons, all lay between the normal limits for white and grey irons. Suitable annealing rendered most of the martensitic alloys capable of being machined. All the nickel-silicon alloys were unstable on heating, though to a less degree than ordinary silicon irons, and liability to corrosion was increased with the lower contents of nickel, and retarded when higher additions of nickel produced an austenitic structure. C. A. KING.

Constitution of silicon-carbon-iron alloys and a new theory of the cast irons. D. HANSON (Iron and Steel Inst., Sept., 1927. Advance copy. 41 pp.).—During investigation into the constitution of ternary alloys of iron containing 0—2% Si and 0—4% C it was shown that, in ternary alloys, graphite and cementite can occur as stable phases either separately or together. The consequences following the two diagrams representing graphite and carbide, respectively, overlapping instead of remaining always one above the other are discussed, and experimental evidence is advanced for the assumption that the iron-carbide and iron-graphite systems intersect at a point on that boundary of the austenite field representing carbon solubility. The theory is consistent with the stability of iron carbide in the range of ordinary steels, the normally-accepted equilibrium diagrams for these requiring no modification in principle, and accounts for the existence of graphite in stable form in alloys, *e.g.*, cast iron, containing more than 2% C and 0·4% Si with the exception of a short range of temperature in the region of 700°, in which range some carbide can exist where the silicon content is low.

C. A. KING.

Quantitative measurement of the cutting power of cutlery. K. HONDA and K. TAKAHASI (Iron and Steel Inst., Sept., 1927. Advance copy. 19 pp.).—The cutting power and durability of sharpness of cutlery was determined by reciprocal motions of the cutting edge under a definite load, on a number of superimposed sheets of paper, the number of sheets cut indicating the relative sharpness. Dulling of the edge with repeated use is represented by a logarithmic curve. Steel containing 1·3% C gave the best results for carbon steels, and the addition of 1% Cr (or W) or 0·5% Mo increased both the initial sharpness and the durability of the blades.

C. A. KING.

Testing machine for repeated impact: effects of repeated impact on Lowmoor iron. J. H. SMITH and F. V. WARNOCK (Iron and Steel Inst., Sept., 1927. Advance copy. 33 pp.).—On a machine designed to apply repeatedly stresses of known energy by means of a falling weight results showed that for a given amount of repeatedly applied tensile shock energy, the total energy to rupture a material is constant and momentum is not an important factor. For a given weight the height of fall and the number of shocks to produce rupture are connected by an equation of the form $H = AN^{-x}$ for a portion of the range, after which the value of x alters. The energy required to produce rupture by a single tensile shock is equal to that required by static means, the elongation being 4%, and reduction in area 3% greater than under static conditions. Elongation is more rapid at the beginning and end of a repeated impact test. It was found that for all areas above approximately 1 sq. in. the amount of energy required to rupture was fairly constant, the average value being 2430 in.-lb., but for areas below 1 sq. in. the total energy varies due to losses by distortion of the striking surfaces. The tests showed the serious source of weakness in abrupt changes of section, *e.g.*, a sharp V groove. C. A. KING.

Corrosion of copper pipes. A. F. DUFTON and F. L. BRADY (Nature, 1927, 120, 367).—Copper water pipes lined with tin may fail by local corrosion when the tin becomes tarnished, since the tarnished lining ultimately becomes electro-positive to copper. The *P.D.* between copper and the lining in water fell from about + 0·4 to —0·1 volt. A. A. ELDRIDGE.

Determination of manganese and magnesium in aluminium alloys. F. MUGRAUER (Chem.-Ztg., 1927, **51**, 658).—After precipitation of the iron by zinc oxide, the manganese is determined by titration with potassium permanganate solution. The filtered solution is acidified with hydrochloric acid, concentrated, and treated with excess of ammonium chloride and ammonia. The magnesium is precipitated by addition of sodium phosphate to the hot solution, and the precipitate ignited and weighed as $Mg_2P_2O_7$. F. R. ENNOS.

Preparation of metallic cobalt from cobaltiferous smelter residues. A. BREMHORST (Metall u. Erz, 1927, **24**, 7—8; Chem. Zentr., 1927, I, 1518).—Residues containing 0·5—5% Co are roasted alone or with sodium chloride, carbon, or sodium sulphate and carbon, whereby the cobalt is converted into sulphide. The product is melted and cast into tall moulds; it separates into three layers, the lowest of which consists of almost pure cobalt sulphide. Metallic cobalt may be obtained by addition of carbon to the charge. A. R. POWELL.

Influence of variables on the structure of electrodeposited copper. A. K. GRAHAM (Amer. Electrochem. Soc., Sept., 1927, **52**. Advance copy. 19 pp.). —Under ordinary conditions of deposition electrolytic copper tends to assume the structure of the base metal even when deposited on smooth, highly polished cathodes. Increase of current density or of temperature results in a coarser deposit with a tendency to "treeing." With a low acidity of the bath the structure consists of fine, broken, twinned crystals, but becomes more conical and columnar with higher acid concentrations. Addition agents, such as aluminium sulphate and gelatin, tend to produce more fibrous deposits, and the effect of the structure of the base metal becomes much less marked. A. R. POWELL.

Enamels for sheet steel. CHI-FANG LAI, also HARRISON and WOLFRAM.—See VIII.

PATENTS.

Production of malleable iron and steel directly out of oxide ore. Production of iron and steel. E. G. T. GUSTAFSSON (E.P. 262,126—7, 26.11.26. Conv., 28.11.25).—(A) The direct reduction of ore in an electric furnace is effected by using a deficiency, amounting to 5—10%, of carbonaceous material in the early part of the process, making up the required reducing agent, together with more ore if desired, towards the end of the reaction. (B) The charge of ore and reducing agent is fed on to the top of the slag layer, which is of sufficient viscosity to sustain the mixture until reduction of the ore is effected before reaching the molten metal below. Heating is concentrated during the smelting process to different layers of the slag bath as required by varying the height of the electrodes. C. A. KING.

Manufacture of malleable iron castings. H. A. SCHWARTZ, Assr. to NAT. MALLEABLE & STEEL CASTINGS Co. (U.S.P. 1,640,674, 30.8.27. Appl., 30.10.24).—In making malleable castings with low silicon content, zirconium is added as a graphitising accelerator. The metal is cast with the maximum quantity of combined carbon, and the casting is annealed to precipitate carbon in finely-divided form. C. A. KING.

Blast-furnace method. F. W. DAVIS, Assr. to S. G. ALLEN (U.S.P. 1,640,485, 30.8.27. Appl., 4.10.24).—In the blast-furnace smelting of iron ore an additional supporter of combustion is introduced into the blast and a reducing agent into the lower end of the shaft. A. R. POWELL.

Production of pig iron from fine ore by smelting in a blast furnace. C. KILIAN (G.P. 440,099, 15.11.24). —Fine iron ore is mixed with sufficient coke to effect the reduction to metal, and the mixture is charged into the throat of the blast furnace so that as it falls down through the furnace the ore is reduced to metal. Just sufficient coke is supplied in lump form to the furnace to melt the reduced metal, and the gases rich in carbon monoxide are taken from the throat of the furnace and passed back through the tuyères. A. R. POWELL.

Manufacture of cast iron and steel. J. M. LARGE (F.P. 609,138, 12.1.26).—A mixture of iron ore and wood charcoal is heated indirectly in a shaft furnace surrounded by a hollow chamber filled with lumps of lime heated at 1800—2000° by means of burning gases. The molten product is discharged from the bottom of the shaft into a second chamber similarly heated, and the metal is siphoned from under the slag layer into a third chamber similarly heated, in which it is carburised as usual. The iron and steel so obtained are free from sulphur and phosphorus. A. R. POWELL.

Apparatus for utilising low-grade iron ore. H. H. HINDSHAW, Assr. to HINDSHAW ENGINEERING & DEVELOPMENT Co. (U.S.P. 1,633,839, 28.6.27. Appl., 24.1.21).—The crushed ore is mixed with peat or other carbonaceous material, and heated in a rotary kiln out of contact with air, the reduced product being separated magnetically. T. S. WHEELER.

Treatment [colouring] of the surfaces of stainless or rustless steels or irons. W. H. HATFIELD and H. GREEN (E.P. 275,781, 30.7.26).—The surface of stainless steel or iron is coloured by immersion in a solution consisting of water 1000 pts., chromic acid 200 pts., sulphuric acid 400—450 pts., preferably at a temperature of 40—60°. C. A. KING.

Manufacture of alloy steel. C. T. EVANS (U.S.P. 1,639,989, 23.8.27. Appl., 18.4.22).—The steel consists of 3·50—7·75% Cr, 10—20% Ni, 0·4—1·0% C, 0·4—2·5% Mn, 0·5—2·5% Si, the remainder being principally iron. F. G. CROSSE.

[Vanadium-iron] alloy. L. JORDAN and G. W. QUICK, Assrs. to UNITED STATES OF AMERICA (U.S.P. 1,638,855, 16.8.27. Appl., 27.12.26).—An iron alloy contains approximately 2—4% C and 10—15% V. F. G. CROSSE.

[Ferrous] casting having a chromium alloy surface. C. B. JACOBS, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,632,704, 14.6.27. Appl., 22.4.21).—Ferrous castings are coated with a layer of chromium alloy resistant to oxidation by lining the moulds with granulated chromium mixed with a binder, *e.g.*, sodium silicate, and pouring the castings at about 1500°. T. S. WHEELER.

Production of magnetic material. L. ZICKRICK, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,632,105,

14.6.27. Appl., 25.3.25).—Magnetic alloys of iron and antimony (2—15%) are claimed, which can be easily reduced to powder. T. S. WHEELER.

Roasting furnace. H. J. HARTLEY, E. J. FOWLER, and D. BAIRD, Assrs. to NICHOLS COPPER CO. (U.S.P. 1,640,988, 30.8.27. Appl., 21.7.26).—In a rotary shaft furnace containing a number of superimposed hearths, each hearth is constructed in removable sections supported by the furnace wall. C. A. KING.

Smelting furnace. T. LEWIN, Assr. to W. LEWIN (U.S.P. 1,640,574, 30.8.27. Appl., 6.7.25).—A smelting furnace consists of an elongated cylindrical shell with removable end plates which may be fastened to collars fixed to the cylinder casing. C. A. KING.

Bright-annealing furnace. SIEMENS-SCHUCKERTWERKE G.M.B.H. (E.P. 262,445, 2.12.26. Conv., 3.12.25). —In a furnace for bright annealing, the space between the furnace body and a protective casing is filled with a gas which will not explode with air or with the protecting gas in the furnace chamber, *e.g.*, a gas of low oxygen content. C. A. KING.

[Flotation] concentration of metalliferous ores. T. R. SIMPSON, Assr. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,632,419, 14.6.27. Appl., 27.5.26. Conv., 9.6.25).—A mixture of an organic protective colloid, *e.g.*, starch, and an alkaline xanthate is of value as a reagent in the froth flotation of sulphide ores. T. S. WHEELER.

Concentration of ores. R. E. SAYRE, Assr. to METALS RECOVERY CO. (U.S.P. 1,640,218, 23.8.27. Appl., 23.7.25).—The mineral in the form of a pulp is subjected to a flotation process in the presence of a thiourethane. F. G. CROSSE.

[Ore] flotation process. E. A. HALL and G. R. SANDERS, Assrs. to SOUTHWESTERN ENGINEERING CORP. (U.S.P. 1,634,615, 5.7.27. Appl., 13.9.26).—The addition of a small quantity of oxalic acid is of advantage in the froth flotation of zinc and lead ores containing carbonaceous and tarry material. T. S. WHEELER.

Controlling the evolution of elemental sulphur [from sulphide ores]. G. N. KIRSEBOM, Assr. to U.S. SMELTING, REFINING, AND MINING CO. (U.S.P. 1,640,837, 30.8.27. Appl., 16.11.25).—To control the evolution of sulphur from sulphide ores while sintering a quantity of arsenic sufficient to combine with the sulphur is added to the charge. A. R. POWELL.

Method of making [nickel-chromium] alloys. J. H. L. DE BATS, Assr. to DE BATS METALS CO. (U.S.P. 1,639,330, 16.8.27. Appl., 24.1.24).—A finely-divided mixture of metals of the chromium group is placed on the surface of unmelted nickel, the nickel is melted, and the unfused mixture of other metals is mechanically mixed with the melt. C. A. KING.

Welding [of nickel]. INTERNAT. NICKEL CO., Assees. of N. B. PILLING and J. G. SCHOENER (E.P. 259,967, 12.10.26. Conv., 19.10.25).—The welding of nickel or nickel alloys is improved if a metal of good reducing power and having a relatively high b.p. is introduced into the molten weld. For this purpose a nickel or nickel alloy electrode is coated with a composition containing titanium (3 pts.) and calcium (1 pt.). C. A. KING.

Refining lead. P. W. DAVIS (U.S.P. 1,640,486—7, 30.8.27. Appl. [A, B], 11.1.26).—(A) Molten lead alloys are stirred with litharge at a temperature between the m.p. of litharge containing the oxide of the alloying element and the m.p. of pure litharge (about 883°). (B) The metallic product obtained by the above treatment is further refined by treatment with fresh litharge at a temperature above its m.p. Both operations are carried out on relatively small charges of alloy. A. R. POWELL.

Refining and separation of metals. H. HARRIS (E.P. 275,344, 7.5.26).—In the treatment of impure lead containing arsenic, tin, and antimony with molten caustic soda the greater part of the antimony remains insoluble when extracted with water. The smaller soluble portion is separated by treating the solution with a reducing agent, *e.g.*, tin, stannous chloride. C. A. KING.

Recovery of copper, zinc, and other metals from solutions containing chlorides and sulphates. J. Y. JOHNSON. From DUISBURGER KUPFERHÜTTE (E.P. 276,200, 18.11.26).—The solution containing copper, iron, and zinc, together with chlorides and sulphates of the alkali metals (*e.g.*, the solution obtained by leaching the product obtained by the chloridising roasting of sulphide ores of copper and zinc), is treated with zinc hydroxide to precipitate copper and iron hydroxides, and the filtrate is treated with zinc dust to remove lead, silver, and the last traces of copper. The clear liquor is cooled to deposit sodium sulphate crystals, calcium chloride is added to remove the remaining sulphate, and the filtrate is treated with lime to precipitate zinc hydroxide for further use in the process and to regenerate calcium chloride. A. R. POWELL.

Apparatus for determining the composition of copper and zinc alloys [brass]. M. POLANYI and S. VON BOGDÁNDY (E.P. 268,306, 17.2.27. Conv., 24.3.26).—Brass is heated in a silica test-tube enclosed in a larger cylindrical silica vessel closed at its lower end and connected to a vacuum pump at its upper end. Heating is effected by lowering the lower end of the large cylinder into a wire-wound electric furnace. At 800—900° zinc is rapidly volatilised from brass *in vacuo* and the residual copper may be weighed (cf. B., 1927, 413). A. R. POWELL.

Metallurgical process. [Treatment of copper concentrate.] W. E. GREENAWALT (U.S.P. 1,633,091, 21.6.27. Appl., 7.9.26; cf. U.S.P. 1,483,056; B., 1924, 340).—Copper concentrate containing rare metals is roasted, most of the copper being then extracted with dilute acid. The solution is treated with sulphur dioxide to reduce the iron present, and the copper recovered by electrolysis. The residue is smelted with pyrites, thus concentrating the residual copper and rare metals into a matte, which is heated to volatilise the sulphur present. The resulting crude copper is electrolytically refined, the rare metals being recovered from the anode slimes. T. S. WHEELER.

Process of hardening copper. W. H. WEISER (U.S.P. 1,638,076, 9.8.27. Appl., 11.10.23).—Pulverised magnesia is injected into a mass of molten copper. H. ROYAL-DAWSON.

Compressed compositions for the production of bearings. C. CLAUS (E.P. 275,444, 13.10.26).—Self-

lubricating bearing blocks are made by compressing a mixture of 85 pts. of finely-divided copper, 9 pts. of tin, 2 pts. of zinc, and 4 pts. of graphite under a pressure of 10,000—50,000 lb./sq. in., and then heating the material in a closed container for 10—20 min. at 760—820°.

C. A. King.

Purifying aluminium and its alloys. Metallbank u. Metallurgische Ges. A.-G. (E.P. 265,563, 13.1.27. Conv., 8.2.26).—Calcium or calcium carbide may be removed from aluminium or aluminium–silicon alloys by melting the metal below a bath of cryolite containing dissolved alumina. The resulting castings are stronger and more ductile, and do not exhibit the coarse grey fracture caused by the presence of calcium.

A. R. Powell.

Producing metal [copper] coatings on articles of aluminium and its alloys. O. Sprenger Patentverwertung Jirotka m.b.H., and B. Jirotka (E.P. 275,828, 27.10.26. Addn. to E.P. 249,971 ; B., 1926, 496).—The bath recommended in the chief patent may be replaced by the following mixture : 25 g. of copper sulphate crystals, 25 g. of sodium carbonate, 25 g. of sodium hydrogen carbonate, and 10 g. of sodium dichromate in 2·5 litres of water. The dichromate may be omitted, but its use leads to a better deposit in a shorter time.

A. R. Powell.

Alumino-solder. M. Torii (U.S.P. 1,640,161, 23.8.27. Appl., 7.8.24).—The solder contains 20—70% Zn, 20—70% Sn, 5—35% Pb, 3—50% Cd, 1—5% Mg, and 1—5% of tin phosphide.

F. G. Crosse.

Protection of magnesium and its alloys. G. Michel (E.P. 249,484, 10.2.26. Conv., 20.3.25).—The cleaned metallic surface is painted or sprayed with phenol or a mixture containing phenol or one of its homologues, whereby a tightly adherent film of a reaction product of phenol and magnesium is formed on the metal. This film may be further protected by a coating of varnish or paint.

A. R. Powell.

Manufacture of refractory metal alloy of high density and high melting point. C. A. Laise (U.S.P. 1,633,258, 21.6.27. Appl., 8.10.25).—Tungsten oxide is reduced in a current of hydrogen and ammonia, the resulting metal being mixed with boron nitride (0·5%), compressed in a mould, and heated at about 900° *in vacuo*. The porous product is immersed in a bath of molten copper, with which it rapidly alloys.

T. S. Wheeler.

Metal pickling. J. H. Gravell (U.S.P. 1,632,833, 21.6.27. Appl., 15.4.26).—Hair, gelatin, or other animal material is destructively distilled, that portion of the distillate which is insoluble in water and soluble in hydrochloric acid being added to metal-pickling baths to reduce action on clean metal surfaces.

T. S. Wheeler.

Production of metals in electric furnaces. E. G. T. Gustafsson (E.P. 252,162, 11.5.26. Conv., 16.5.25).—To protect the refractory bottom of an electric furnace during the interval between tapping and re-charging, a quantity of slag, which may be taken from the previous refining, is introduced into the furnace to act as a protective layer.

C. A. King.

Cupola furnace. A. Poumay (U.S.P. 1,640,251, 23.8.27. Appl., 16.4.25. Conv., 18.4.24).—See E.P. 232,630 ; B., 1926, 132.

Process of rust-proofing [iron]. M. Green and H. H. Willard, Assr. to Parker Rust-Proof Co. (U.S.P. 1,639,694, 23.8.27. Appl., 10.5.26).—See E.P. 273,168 ; B., 1927, 658.

Manufacture of sound silicon-iron. V. B. Browne (E.P. 276,190, 25.10.26).—See U.S.P. 1,627,269 ; B., 1927, 448.

Manufacture of alloys containing gold. General Plate Co., Assees. of V. D. Davignon (E.P. 257,891, 25.5.26. Conv., 4.9.25).—See U.S.P. 1,557,431 ; B., 1926, 63.

Process of metallising ores and recovery of metals and by-products. D. M. Crist (E.P. 275,791, 10.8.26).—See U.S.P. 1,595,454 ; B., 1926, 984.

Regeneration of metal catalysts contaminated by sulphur. E. A. Prudhomme, Assr. to Soc. Internat. des Proc. Prudhomme (U.S.P. 1,640,668, 30.8.27. Appl., 8.5.26).—See E.P. 238,805 ; B., 1925, 834.

Recovery of copper, zinc, and other metals from solutions containing chlorides and sulphates. K. Mattenklodt and H. Schramm, Assrs. to Duisburger Kupferhütte (U.S.P. 1,639,610, 16.8.27. Appl., 17.11.26. Conv., 9.4.25).—See E.P. 276,200 ; preceding.

[Reinforced] lead columns or pipes etc. [for acids]. Soc. Belge de l'Azote (Soc. Anon.) (E.P. 263,881, 3.1.27. Conv., 4.1.26).

[Electric] bright-annealing furnace [with liquid sealing]. Siemens-Schuckertwerke G.m.b.H. (E.P. 262,462, 3.12.26. Conv., 5.12.25).

Fuel for welding (U.S.P. 1,628,067).—See II.

Luminous enamels (F.P. 610,454). **Enamelling sheet iron** (F.P. 610,645).—See VIII.

Electrodeposition of chromium (G.P. 440,196).—See XI.

XI.—ELECTROTECHNICS.

"Knowles'" plant for electrolysis of water. Q. Sestini (Giorn. Chim. Ind. Appl., 1927, **9**, 318—320).—A short description is given of the construction of this cell in which 15% sodium hydroxide is used as electrolyte, and which is said to deliver 99·5% O_2 and 99·8% H_2. Operating characteristics are given for a cell consuming 4500 amp. at 2·5 volts.

R. W. Lunt.

Electrical insulating materials. A. Günther-Schulze (Z. Elektrochem., 1927, **33**, 360—369).—A lecture.

High-frequency induction melting. Campbell. **Tempering of steels.** Matsushita and Nagasawa. **Structure of electrodeposited copper.** Graham.—See X.

Patents.

Electric furnace. J. Kelleher, Assr. to Harper Electric Furnace Corp. (U.S.P. 1,637,486, 2.8.27. Appl., 17.7.25).—A carbonaceous resistor extends longitudinally through a furnace chamber having end and

side walls of refractory silica material, and is surrounded by an inner lining of carbonaceous material forming a resistor chamber. A packing between the inner lining and outer wall is sufficiently thick to prevent entrance of silicon vapour from the furnace chamber walls into the resistor chamber. J. S. G. THOMAS.

Electric resistance furnace. F. KRUPP A.-G. (E.P. 257,896, 19.7.26. Conv., 4.9.25).—In an electric resistance furnace at least one receptacle for receiving articles to be treated rests on a roller system, *e.g.*, through an intermediate member, for which a track is provided in the heating chamber and to which similar tracks for entrance and exit of the receptacle are attached. The heating chamber consists of a chromium–nickel steel or chromium–steel alloy. J. S. G. THOMAS.

Preparation of an electron-emitting cathode. F. HOLBORN, Assr. to HAZELTINE CORP. (U.S.P. 1,639,698, 23.8.27. Appl., 14.1.26).—An alkaline-earth metal "sputtered" in pyridine is electrically deposited on a conductor, which is then heated to evaporate the pyridine. J. S. G. THOMAS.

Incandescence cathode. H. WADE. From N.V. PHILIPS' GLOEILAMPENFABR. (E.P. 275,449, 27.10.26. Addn. to E.P. 274,981; B., 1927, 754).—The prior patent is modified in that the metal wire wound helically upon a refractory metal core is composed of nickel. J. S. G. THOMAS.

[Filament supports of] electric incandescence lamps. GEN. ELECTRIC CO., LTD., and C. J. SMITHELLS (E.P. 275,735, 14.5.26).—Filament supports, except those parts in contact with the filament, are made of or coated with chromium, manganese, or uranium, but preferably chromium, which serves to decompose any oxidising material, and more especially water vapour, contained in the lamp. J. S. G. THOMAS.

Preparing positive electrodes of storage batteries. A. CELLINO, Assr. to CENTRAL BATTERY CORP. (U.S.P. 1,640,922, 30.8.27. Appl., 15.6.16. Renewed 8.1.27).—An electrode formed of a mixture of an oxide of lead, manganese peroxide, and ammonium sulphate is treated with sulphuric acid to dehydrate the active material. J. S. G. THOMAS.

Accumulator plate. W. HADDON and J. M. BURNETT (E.P. 275,392, 7.7.26).—Accumulator plates are pasted with a mixture containing sulphur dissolved in a solution of sodium hydroxide and the lower oxides of lead. The plate is, after an interval of 2—3 days, immersed in a mixture of lead peroxide and sulphuric acid or a soluble sulphate and formed in an acid bath. J. S. G. THOMAS.

Accumulators containing lead dioxide, zinc, and sulphuric acid. A. MIOLATI, Assr. to R. LEPRESTRE (U.S.P. 1,632,307, 14.6.27. Appl., 22.4.24. Conv., 3.5.23).—The efficiency of such accumulators is increased by having the positive plate of lead and lead dioxide, the electrolyte of sulphuric acid, and the negative plate of lead alloyed with 2—3% of zinc, all materials being pure. T. S. WHEELER.

Dry cell. V. YNGVE, Assr. to NATIONAL CARBON CO., INC. (U.S.P. 1,634,006, 28.6.27. Appl., 12.1.25).—The efficiency of Leclanché dry cells is increased by grinding together the mixture of manganese dioxide, graphite, and ammonium chloride used, and then adding the electrolyte. T. S. WHEELER.

[Depolariser for] galvanic cells. C. F. BURGESS LABORATORIES, Assees. of B. K. BROWN (E.P. 255,067, 7.7.26. Conv., 7.7.25).—The depolariser used in a galvanic cell of the Leclanché type consists of solid oxides of carbon, *e.g.*, graphitic oxide, prepared in accordance with E.P. 255,068 (B., 1927, 17) mixed, if desired, with manganese dioxide. J. S. G. THOMAS.

Dielectric covering for electric wires, cables, etc. P. ALLMAN, H. N. MORRIS, and L. H. MARLOR (E.P. 275,304, 30.4.26).—Electric wires, cables, etc. are covered by deposition with a rubber–cellulose coating obtained from a combined solution of rubber and cellulose in one or more of the volatile hydrogenated products of aromatic hydrocarbons, or their hydroxy-compounds or isomerides, or esters of these, *e.g.*, *cyclo*hexanone, butyl acetate, with or without non-volatile solvents or softening media. J. S. G. THOMAS.

Electrolytic apparatus [for electrolysis of water]. W. G. ALLAN (E.P. 275,785, 4.8.26).—Apparatus for the electrolysis of water comprises a group of cells, a stand pipe, and a pair of return-flow manifolds communicating with a tank, a tube communicating between each manifold and each cell of a group, a second pair of manifolds connected, respectively, by conduits to the anode and cathode sides of each cell, conduits connecting, respectively, the anode and cathode manifolds with chambers of the tank, and a gas outlet from each chamber of the tank. J. S. G. THOMAS.

Electrolytic diaphragm cells. J. BILLITER, and SIEMENS & HALSKE A.-G. (Austr. P. 105,356, 23.2.25).—In a diaphragm cell excess pressure on the anode side is practically uniformly distributed by filling the cathode chamber with electrolyte over the whole surface of the diaphragm, whilst the difference of level of anolyte and catholyte can be so great that the electrolyte traverses the diaphragm at such a rate that 30%, or even 60%, of the salt is decomposed. J. S. G. THOMAS.

Anode [for electrolysis]. E. C. R. MARKS. From A. P. MUNNING & CO. (E.P. 275,359, 26.5.26).—The crystals, *e.g.*, of nickel, forming the major active portion of an aonde are disposed substantially normal to the main path of current travel in an electrolytic bath. J. S. G. THOMAS.

Electrolytic decomposition of water. VEREIN FÜR CHEM. U. MET. PROD. (G.P. 440,335, 31.1.26).—Alkali hydroxide solution produced by the electrolysis of alkali chloride solution by the mercury process is used as electrolyte in the electrolytic decomposition of water. L. A. COLES.

Electrodeposition of chromium. SIEMENS & HALSKE A.-G., Assees. of M. HOSENFELD and H. WALDE (G.P. 440,196, 3.7.25).—In the electrodeposition of chromium from chromic acid baths, the anode consists of one or more lead plates lying on the floor of the cell and covered loosely with powdered chromium, preferably electrolytically produced. A. R. POWELL.

Prevention of oxidation of oil in transformers. J. KUBLER, Assr. to A.-G. BROWN, BOVERI, & CIE. (U.S.P.

1,635,332, 12.7.27. Appl., 2.9.26. Conv., 24.9.25).—An atmosphere of carbon dioxide is maintained above the surface of the oil by an apparatus working on the Kipp principle. T. S. Wheeler.

Electrochemical production of solid oxides of carbon [graphitic oxide]. C. F. Burgess Laboratories, Inc., Assees. of B. K. Brown, O. W. Storey, and G. T. Collinson (U.S.P. 1,639,980—2, 23.8.27. Appl., 7.7.25).—See E.P. 255,068; B., 1927, 17.

Galvanic cell. C. F. Burgess Laboratories, Inc., Assees. of B. K. Brown (U.S.P. 1,639,983—5, 23.8.27. Appl., 7.7.25).—See E.P. 255,067; preceding.

Decolorising carbon (U.S.P. 1,634,477—80). **Metallic carbon** (U.S.P. 1,637,052). **Acidity in transformer oils** (U.S.P. 1,633,956).—See II.

Preserving wood (G.P. 439,523).—See IX.

Magnetic alloys (U.S.P. 1,632,105).—See X.

XII.—FATS; OILS; WAXES.

Drying of fatty oils. Gas-coagulation of linseed oil. P. Slansky (Chem. Umschau, 1927, **34**, 148—151).—The author claims priority for the view that the coagulation processes leading to the solidification of drying oil films are necessarily subsequent to purely chemical reactions. The view that drying may occur entirely through colloidal changes (supported by Auer and Wolff) is criticised on theoretical grounds. On keeping linseed oil films of various thickness *in vacuo* for 16 days, under conditions precluding any infiltration of air, negligible changes in viscosity, refractive index, iodine value, and weight are found to occur. From consideration of these results the validity of Auer's "drying *in vacuo*" experiments and the gas-coagulation theory he deduces therefrom is called into question. S. S. Woolf.

Oil of *Salvia spinosa*. S. Berlingozzi and G. Di Mase (Rend. Accad. Sci. Fis. Mat. Napoli, 1926, [iii], **31**, 156—157).—See B., 1926, 887.

Yellowing of drying-oil films. Morrell and Marks.—See XIII.

Patents.

Manufacture of grease. J. McKee, Assr. to Sun Oil Co. (U.S.P. 1,637,703, 2.8.27. Appl., 24.8.25).—A mixture of 5—50% of hard coconut oil soap and 95—50% of a paraffin base mineral oil is heated at above 232°, forming a grease of high b.p.

H. Royal-Dawson.

Apparatus for the treatment [to separate kernels and pulp from nuts] of oil-containing fruits [oil-palm]. F. Krupp Grusonwerk A.-G. (E.P. 267,092, 25.1.27. Conv., 4.3.26).

Apparatus for hydrogenation (E.P. 274,952).—See I.

Removal of odour from oils (G.P. 439,615).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Yellowing of drying-oil films. R. S. Morrell and S. Marks (J. Oil Col. Chem. Assoc., 1927, **10**, 186—200).—The changes in colour of various white paint films kept in the dark in a moist atmosphere at ordinary temperatures were observed, using yellow and red Lovibond tintometer glasses with a freshly-made zinc oxide–poppyseed oil paint as standard. Experimental details are quoted, and the extent of yellowing of the films after 1, 2, 3, and 7 months is recorded. The yellowing with raw drying oils was less than with prepared bleached linseed oil, whilst stand oil was superior to raw oil, closely approaching the quality of walnut and poppyseed oil, which showed the least yellowing. White lead, which in general caused greater yellowing than did zinc oxide, was unsatisfactory with the bleached oils. Of the various white pigments tested, anhydrous titanium dioxide gave the best results. The oxidation of β-elæostearic glyceride is being studied, with the object of isolating the yellowing principle, and a preliminary account of the investigation is given. From the results to date, it is deduced that the property of yellowing in the dark is probably connected with the presence of hydroxyl groupings derived from the peroxide group contiguous to the carboxyl end of the molecule, and may be induced by traces of water or the elements of water in a pigment. S. S. Woolf.

Pigments for colouring Portland cement mortar. J. E. Foster (Chem. Met. Eng., 1927, **34**, 487—488).—Various pigments used for colouring cement were tested for tinting value, stability to light, and their effect on the strength of the cement. It was concluded that in no case was a greater addition than 20% of any advantage. Metallic oxides used were iron oxides for yellow, buff, red, brown, and black, chromium oxide for green, and manganese dioxide for black. These all proved permanent to light, and an admixture up to 20% did not reduce strength. Ultramarine was also satisfactory. Carbon black seriously reduced the cement strength. Zinc chromate and cadmium lithopone failed owing to impermanence. The colour of the sand used is not without importance. When possible it should harmonise with the pigment. C. Irwin.

Patents.

Production of [wood] paints and stains. G. W. Carver (U.S.P. 1,632,365, 14.6.27. Appl., 29.1.25. Cf. U.S.P. 1,541,478; B., 1925, 601).—Clay containing a high percentage of iron and free from alkali is heated with iron turnings and a mixture of hydrochloric and sulphuric acids, the suspension obtained being treated with potassium ferricyanide and nitric acid. The blue pigment formed is of value in the treatment of wood.

T. S. Wheeler.

Production of lead compounds of chromic acids. A. Nathansohn (U.S.P. 1,633,948, 28.6.27. Appl., 19.11.24. Conv., 18.9.24).—Lead ores are heated with aqueous alkali or alkaline-earth chloride solutions, the resulting solutions being treated with potassium dichromate or the like to precipitate lead compounds of chromic acids of value as pigments. T. S. Wheeler.

Manufacture of precipitated antimony [penta]-sulphide. R. E. Stark, Assr. to Stibium Products Co. (U.S.P. 1,633,754, 28.6.27. Appl., 26.12.22; cf. U.S.P. 1,414,836—7; B., 1922, 474 a).—An oxide of antimony, sulphur, and a compound capable of forming a soluble compound containing antimony, *e.g.*, calcium oxide, are heated with water under pressure at 155°, the resulting solution being treated with hydrochloric

acid, with or without addition of antimony pentachloride, to precipitate antimony pentasulphide.

T. S. Wheeler.

Non-inflammable varnish. H. C. P. Weber, Assr. to Westinghouse Electric & Manuf. Co. (U.S.P. 1,635,567, 12.7.27. Appl., 5.7.22).—A mixture of carbon tetrachloride and chlorobenzene is added to varnish solutions in place of inflammable solvents. To inhibit formation of acidity on drying the inclusion of a small proportion of aniline or the like is preferred.

T. S. Wheeler.

Phenolic condensation product. A. L. Brown, Assr. to Westinghouse Electric & Manuf. Co. (U.S.P. 1,632,113, 14.6.27. Appl., 7.4.22; cf. U.S.P. 1,633,976; following abstract).—The addition of a drier, *e.g.*, "tox tungate" (a mixture of lead and manganese resinates and tungates), to the ingredients employed in the manufacture of cresol–formaldehyde resins is claimed.

T. S. Wheeler.

Manufacture of a flexible composite article. A. L. Brown, Assr. to Westinghouse Electric & Manuf. Co. (U.S.P. 1,633,976, 28.6.27. Appl., 24.9.20. Renewed 8.11.26; cf. U.S.P. 1,632,113; preceding abstract).—The addition of a drier, *e.g.*, "tox tungate," and of a polymerisable vegetable oil, *e.g.*, tung oil, to the ingredients employed in the manufacture of phenol–formaldehyde resins gives rise to a product of increased flexibility.

T. S. Wheeler.

Resinous reaction product of urea and formaldehyde. F. Lauter, Assr. to Rohm & Haas Co. (U.S.P. 1,633,337, 21.6.27. Appl., 28.7.25).—Formaldehyde is condensed with urea in presence of glycerol, or of a non-aqueous solvent, *e.g.*, benzene, to give resinous products insoluble in water.

T. S. Wheeler.

Manufacture of sulphur–phenol resin. C. Ellis (U.S.P. 1,636,596, 19.7.27. Appl., 30.12.22).—A phenol (1 mol.), sulphur (more than 3 atoms), and an alkaline catalyst, *e.g.*, sodium hydroxide, are heated at about 170°.

T. S. Wheeler.

Barium carbonate and sulphydrate (U.S.P. 1,634,338).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Ageing of soft rubber goods. R. F. Tener, W. H. Smith, and W. L. Holt (U.S. Bur. Standards, Tech. Paper No. 342, 1927, **21**, 353—384).—Four different types of vulcanised mixed rubber containing (A) rubber 90, sulphur, 10; (B) rubber 100, sulphur 4, diphenylguanidine 0·5, zinc oxide 4, whiting 50; (C) rubber 100, sulphur 6, litharge 10, whiting 50; and (D) rubber 100, hexamine 1, sulphur 6·53, zinc oxide 18·75, gas black 34·85, mineral rubber 10·9, palm oil 6·53, and reclaimed rubber 38·1 pts., respectively, were submitted to ageing tests under various conditions, the progress of the change being followed by tensile tests. The rate of deterioration was greater in oxygen than in nitrogen, air occupying an intermediate position. All the compounds deteriorated more rapidly when exposed to light, but (D) was much less sensitive than (B), which was greatly improved, however, by the introduction of a little red iron oxide or of carbon black. Sunlight caused deterioration even in the absence of air, a hard brittle coating being formed, particularly on (A) and (B); the coating, which contained more combined sulphur than the inside rubber, was insoluble in acetone and benzine and appeared to protect against further deterioration. Mixing (B) was most resistant to heat, whereas (C) was most sensitive; all four compounds appeared to be most susceptible at a critical temperature between 80° and 90°. The degree of vulcanisation had greatest influence on (A) and least on (B) and (D). For (C) and (D) the rate of ageing was reduced to approximately one half by a fall of 10° from 90° or 80°. The relative order of ageing in accelerated tests was in good agreement with that in tests with exposure away from sunlight, and the statement that 24 hrs. in air at 70° are equivalent to about 6 months of natural ageing is approximately correct; accelerated tests, however, showed no relation to tests with exposure to sunlight. In outdoor tests, (B) showed greatest deterioration in physical properties and greatest increase in acetone extract, whilst (D) was least in these features; (C) was intermediate in both features, whereas with (A) these features showed no interdependence. In the absence of sunlight (B) deteriorated least and showed no increase in acetone extract; (C) and (D) deteriorated similarly, showing corresponding increases in acetone extract, whilst (A) did not show increases in acetone extract corresponding with the physical depreciation.

D. F. Twiss.

Constitution of caoutchouc. R. Pummerer (Kautschuk, 1927, 233—237).—By prolonged mechanical working, even with exclusion of air, rubber can be so disaggregated that it becomes entirely soluble in ether. Disaggregation can also be effected by dissolving in benzene and re-precipitation, the change being expedited by the presence of basic or acidic substances, *e.g.*, piperidine or acetic acid, in the benzene. The change cannot be a purely chemical one, but is probably to be attributed to a disruption of molecular aggregates. The process of disaggregation is reversible and "sol" rubber isolated from solution may gradually undergo spontaneous conversion into the ether-insoluble "gel" condition; such aggregation is regarded as the result of the grouping of molecules into a colloid-chemical or crystalline arrangement. Cryoscopic measurements in camphor, benzylidenecamphor, benzoic acid, or menthol give results between 1100 and 2500 for the mol. wt. of rubber. Sol and gel rubber in menthol give similar values, viz., 1200—1600 for solutions exceeding 2% in concentration; at 1% or lower, however, the apparent mol. wt. of rubber falls to about 600. Although the osmotic pressure of rubber in benzene indicates a mol. wt. between 30,000 and 50,000, rubber can be dialysed slowly from its benzene solution through a collodion membrane. The results suggest that the molecular nuclei in rubber which are capable of aggregation contain approximately 8 isoprene groups. From their absorption in the ultra-violet, however, neither sol nor gel rubber can have as high a proportion as one pair in 500 of its double linkings in conjugation, or any appreciable proportion of radicals with tervalent carbon. It appears probable, therefore, that the molecular nuclei in rubber involve a cyclic structure.

D. F. Twiss.

Oxidation of rubber. M. Leon and W. N. Lister

(J.S.C.I., 1927, 46, 220—224 T).—In comparative oxidation experiments with films of fine hard Para, soft Para, pale crêpe, brown crêpe, and smoked sheet rubber on the inside of a flask at 100° containing oxygen, the first-named is found to interact with less oxygen than are the others. Moderate mastication of pale crêpe rubber causes a slight increase in the resistance to oxidation, but the over-masticated rubber oxidises very rapidly, although the final quantity of oxygen absorbed is less. A film of rubber made from latex oxidises less rapidly than pale crêpe rubber, but its resistance is decreased by extraction with water. Removal of the natural resins from smoked sheet or pale crêpe causes a great increase in the rate of oxidation, whereas introduction of such resins or increase in the proportion of natural, protein-rich, insoluble matter increases the stability. Dragon's blood resin, dinitrobenzene, and quinol exert a protective action; benzoic acid, resorcinol, and traces of copper chloride accelerate the oxidation. Petroleum jelly and castor oil, which decrease the permeability of rubber to gases, cause an initial retarding effect and later acceleration. D. F. Twiss.

Oxidation of rubber from the scientific and technical point of view. F. Kirchhof (Kautschuk, 1927, 239—245, 256—261).—A review of the subject covering the influence of natural and ultra-violet light on the phenomena of ageing, and of metallic salts on the depolymerisation and oxidation of rubber; the effect of increased temperature on oxidation in air and in oxygen is also discussed. It is suggested that the natural resins in rubber protect the rubber from oxidation because they are chemically more unsaturated, and so combine preferentially with oxygen.

D. F. Twiss.

Recent röntgenographic researches on rubber and related substances. E. A. Hauser [with M. Hünemörder and P. Rosbaud] (Kautschuk, 1927, 228—230).—A preliminary account of results obtained. Unstretched frozen rubber can show Debye-Scherrer circles; on stretching, these lose in intensity and point interference develops. All rubber samples, indeed, which are turbid or opalescent show Debye-Scherrer circles. Rubber also can be extended slowly without loss of its isotropic character, whereas on rapid extension it will give rise to interference phenomena. The sol and gel constituents of rubber, separable by ether, also show interference phenomena when stretched, although it is necessary to keep the sol rubber in an atmosphere of carbon dioxide. The interference phenomena with gutta-percha and balata stretched at 35—40° are practically identical, although differences are observed in the results with the unstretched material.

D. F. Twiss.

Structure of gutta-percha and balata. H. Miedel (Kautschuk, 1927, 230—232).—The behaviour of gutta-percha when stretched under various conditions is described. The hydrocarbons of rubber and of gutta-percha or balata show approximately the same mol. wt. in camphor, and their light absorption in the ultra-violet region gives no indication of additional ring formation in either. From the greater ease of crystallisation of the hydrocarbon of gutta-percha or balata and its apparent resistance to resolution by fractionation, it is probable that this hydrocarbon is more homogeneous than caoutchouc in chemical composition.

D. F. Twiss.

Patents.

Manufacture of articles from ebonite and like material. A. Fraser, and Rissik, Fraser & Co., Ltd. (E.P. 273,355, 2.2. and 2.11.26).—An ebonite article of "good fracture" is produced from undercured ebonite dust (*e.g.*, with a coefficient of vulcanisation of 25—26), which has been previously warmed at about 130°, by moulding under a pressure upwards of about 500 lb./sq. in. and at a temperature near 220—250° for from 3 to 10 min. The moulding chamber is cooled before the article is removed. D. F. Twiss.

Accelerating the vulcanisation of rubber. L. B. Sebrell, Assr. to Goodyear Tire & Rubber Co. (U.S.P. 1,635,193, 12.7.27. Appl., 16.9.26. Cf. U.S.P. 1,604,199;, B., 1927, 52).—Vulcanisation is effected in the presence of a 1-mercaptodimethylbenzthiazole or a derivative. D. F. Twiss.

Production of substances containing rubber and cellulose derivatives. P. Allman, H. N. Morris, and L. H. Marlor (E.P. 274,968, 30.4.26).—Rubber is dissolved in one or more of the volatile hydrogenation products of aromatic hydrocarbons or of hydroxy-, ketonic, or acyloxy-derivatives of such hydrocarbons; non-volatile softening agents may also be present. A solution of a cellulose ester or of celluloid is then introduced in a simple or mixed solvent, *e.g.*, acetone, in which rubber is not soluble. D. F. Twiss.

Process of rubber vulcanisation. C. O. North, Assr. to Rubber Service Laboratories Co. (U.S.P. 1,634,336, 5.7.27. Appl., 27.8.26).—The product formed by the interaction of aniline, acraldehyde, and heptaldehyde is of value as an accelerator.

T. S. Wheeler.

[Accelerators for] rubber vulcanisation. S. B. Molony and Y. Nikaido, Assrs. to C. W. Brown, W. L. Clause, and E. Pitcairn (U.S.P. 1,632,617, and 1,632,666—7, 14.6.27. Appl., [A] 12.4.20. Renewed 18.12.26; [B, C], 25.9.26).—(A) The use of aromatic disubstituted dithiocarbamates, *e.g.*, phenyl phenyldithiocarbamate, is claimed. (B) Carbon disulphide (10 pts.), aniline (20 pts.), and ethyl alcohol (5 pts.) when mixed and exposed to a current of air yield unstable *phenylammonium phenyldithiocarbamate*, which is treated with aqueous alcoholic zinc sulphate solution to give *zinc phenyldithiocarbamate*. (C) Phenylthiocarbimide reacts with methylmercaptan to give *methyl phenyldithiocarbamate*. T. S. Wheeler.

Vulcanisation of rubber. J. Teppema, Assr. to Goodyear Tire & Rubber Co. (U.S.P. 1,637,790—1, 2.8.27. Appl., [A] 12.2.26; [B] 3.11.26).—Rubber is vulcanised in the presence of (A) a halogen derivative of a mercaptobenzthiazole, or (B) a metallic salt of the compound used in (A), as accelerator. D. F. Twiss.

Vulcanisation of rubber substances. Grasselli Chemical Co., Assees. of I. Williams and W. B. Burnett (E.P. 249,113, 8.3.26. Conv., 13.3.25).—The condensation products obtainable from an aldehyde and a nitrogenous base generally have greatest accelerating power

when the molecular proportion of these ingredients lies between 2 : 1 and 7 : 1. Within this range, the most powerful accelerators of vulcanisation are obtained by the condensation of primary amines, *e.g.*, aniline, *n*-butylamine, ethylamine, and *o*-tolyldiguanide, with $\alpha\beta$-saturated aldehydes, *e.g.*, propaldehyde, *n*-butaldehyde, and heptaldehyde (cf. E.P. 263,853 ; B., 1927, 757). D. F. Twiss.

Plastic material. A. R. Kemp, Assr. to Western Electric Co., Inc. (U.S.P. 1,638,535, 9.8.27. Appl., 12.7.24).—Material with the thermoplastic properties of gutta-percha is obtained by submitting a mixture of rubber with 8—16% S to a sufficiently high temperature. D. F. Twiss.

Treatment [purification] of rubber latex. E. Hopkinson and W. A. Gibbons, Assr. to Naugatuck Chemical Co. (U.S.P. 1,632,759, 14.6.27. Appl., 9.6.25). —Crude rubber latex is mixed with a small quantity of calcium polysulphide, being then easily filterable. The residue is further purified by treatment with a proteolytic enzyme, *e.g.*, trypsin, followed by extraction with acetone. T. S. Wheeler.

Manufacture of artificial indiarubber. V. Villa (E.P. 273,029, 1.4.26).—Vegetable or animal oil, *e.g.*, colza oil (75—85 pts.), is subjected to the action of sulphur chloride (6—12 pts.) in a closed vessel with exclusion of air, preferably between 0° and 15°. As catalyst (3—8 pts.) is used a rubber " substitute " produced from oil and sulphur chloride, from which the chlorine has been removed by dissolving in a liquid, *e.g.*, benzene, at a temperature preferably above 40°. It is thus possible to obtain a product containing only a small proportion of sulphur chloride without any previous oxidation or sulphuration of the oil. D. F. Twiss.

Production of rubber articles. S. G. S. Dicker. From Rubber Latex Res. Corp. (E.P. 273,991, 14.2.27). —A jet of latex, containing fibre or fabric, is superficially coagulated by contact with acid, and is then allowed to undergo internal coagulation. The material is then pressed, washed, and dried. With approximately 25% or less of cotton fibre in random arrangement in 75% of rubber, the product has the character of a reinforced rubber material. D. F. Twiss.

Reclaiming rubber. Mixing a soluble filler with rubber. C. H. Campbell, Assr. to American Glue Co. (U.S.P. 1,640,817—8, 30.8.27. Appl., [A, B] 22.10.26).—(A) The albuminate of a metal is added to old rubber prior to " devulcanisation." (B) In mixing a soluble filler with rubber, with the use of heat, a fine spray of water is applied to the mass until the filler has dissolved and thorough incorporation has been effected. Moisture is subsequently expelled from the mass. D. F. Twiss.

Method of forming sponge rubber materials [of great length]. E. C. R. Marks. From Featheredge Rubber Co. (E.P. 273,223, 11.4.27).

XV.—LEATHER ; GLUE.

Tannin of *Heuchera Americana*, Linn. J. C. and B. L. de G. Peacock (J. Amer. Pharm. Assoc., 1927, **16**, 729—737, and Amer. J. Pharm., 1927, **99**, 471—482). —The filtered, cold aqueous extract of the powdered drug was extracted with ethyl acetate and the extracted matter purified by repeatedly re-dissolving in water, filtering, and re-extracting with ethyl acetate. An aqueous solution of the tannin obtained yielded only small extracts when treated successively with light petroleum, chloroform, benzol, and ether. The tannin, recovered from this aqueous solution by extraction with ethyl acetate, differs in appearance and solubility from the phlobaphen previously reported (Amer. J. Pharm., April, 1891), and conforms to the description given in the U.S. Pharmacopœia X for gallotannic acid, though it differs from this substance in some of the reactions described. *E.g.*, it gives precipitates with bromine water and with calcium hydroxide, dissolves readily in *iso*propyl alcohol, and yields with mineral acids a red insoluble phlobaphen together with traces of a crystalline substance which appears to be present in the drug as such, and not formed by hydrolysis. Under certain conditions of hydration the cold water-soluble tannin of the drug is soluble in ether, and the blue colour given by an ether extract with ferric salts is not due to gallic acid, as was originally supposed. Exhaustive treatment with hide powder of a cold water infusion failed to remove all substances yielding a blue colour with ferric chloride, but the coloration, unlike that due to gallic acid, was transient. The results of successive extraction of the drug with chloroform, ether, ethyl acetate, and acetone are described. Ethyl acetate (though an efficient extractive for aqueous infusions) removes very little tannin or phlobaphen from *Heuchera*, but acetone extracts all forms of tannin. Phlobaphen production shows that the tannin of *Heuchera Americana* is not gallotannic acid. C. O. Harvey.

Action of oxalic and hydrochloric acids on vegetable-tanned leathers. D. Woodroffe (J. Soc. Leather Trades' Chem., 1927, **11**, 251—255).—East Indian tanned goatskins were soaked in 1·25% and 4·0% solutions of oxalic acid for 30 min., washed, dried out, and samples tested for strength and stretch. There was only a slight diminution in strength with the stronger solution. The immediate weakening effect of an oxalic acid solution on leather was not serious. Similar tests with solutions of hydrochloric acid showed 35% lower strength by treatment of the leather with the stronger acid, but with the weaker solution of acid it was much lower than that of the leathers treated with the solutions of oxalic acid. Hydrochloric acid has a much more deleterious effect than oxalic acid on vegetable-tanned leathers. D. Woodroffe.

Action of hæmatin and hæmatin-ammonia solutions on vegetable-tanned leathers. D. Woodroffe and A. Meadows (J. Soc. Leather Trades' Chem., 1927, **11**, 262—264).—Pieces of East Indian tanned sheepskin and sumach-tanned skiver were soaked in 200 c.c. of water containing 1, 4, and 8% of hæmatin, respectively, with and without an addition of ammonia, for 20 min., struck out, dried out, and their tensile strength was determined. The results showed a distinct weakening effect by the hæmatin and ammonia solutions and by the stronger hæmatin solutions. The weakening effect was proportionately greater on skiver and thin leathers, whichs eems to indicate that it is the grain which

is most affected. Excessive amounts of hæmatin are deleterious on leather, probably owing to their tannin content. D. WOODROFFE.

Action of hæmatin and iron salts on leather and fabric. D. WOODROFFE and D. B. GILBERT (J. Soc. Leather Trades' Chem., 1927, **11**, 259—262).—Pieces of fabric and chrome-tanned goatskin, when treated with solutions of hæmatin, alone and mixed with ferrous sulphate and with ammonia, respectively, dried out, and tested for strength and stretch, showed no definite weakening effect by 1% or 2% solutions of hæmatin. D. WOODROFFE.

Report of the committee on the revision of the official method for sampling tanning materials. (J. Amer. Leather Chem. Assoc., 1927, **22**, 452—458).

Report of the committee on a provisional method for the separation of pyrogallol and pyrocatechol tannins for 1926—1927 (J. Amer. Leather Chem. Assoc., 1927, **22**, 470—471).

Tannery waste disposal. W. HOWALT and E. S. CAVETT (Proc. Amer. Soc. Civil Eng., 1927, I, 1675—1712).

PATENTS.

Treatment of skins, *e.g.*, hare skins. DELAHAYE, RACHET, & CIE. (F.P. 596,198, 12.7.24).—After the skins have been bated they are placed for 24 hrs. in a solution of sodium carbonate, bicarbonate, borate, chromate, dichromate, hypochlorite, hyposulphite, or any organic salt, or potassium hydroxide (*d* 1·075), then immersed in a sodium sulphide solution, unhaired, washed, and immersed in a second solution of sodium sulphide and excess sodium hydroxide until the mercuric sulphide stains have disappeared, after which the pelts can be tanned. D. WOODROFFE.

Depilating and preserving hides and skins. E. BOHON, E. MAILLIARD, and P. MAILLIARD (F.P. [A] 590,901, 27.2.24; [B] 606,214, 17.2.25; [C] 612,583, 6.7.25).—(A) Soap is heated, phenol and caustic soda or other alkali or alkaline-earth hydroxide are added, and the diluted mixture is boiled. By immersing the skins in the liquor the hair is quickly loosened. (B) The phenol may be replaced by its derivatives or homologues, *e.g.*, tricresol, and the caustic soda by other bases or by a salt having an alkaline reaction. (C) The skins are dipped in a solution of equal parts of resorcinol and a zinc salt, *e.g.*, zinc sulphate, which preserves them against putrefaction and coagulates their albumin. D. WOODROFFE.

Soaking hides. E. BOHON, E. MAILLIARD, and P. MAILLIARD (F.P. 612,409, 30.6.25).—The hides are soaked in a solution of starch, lime, calcium phenoxide, and sodium hydroxide which is effective in half the time required by other means. D. WOODROFFE.

[Manufacture of] glue. W. R. LONG (U.S.P. 1,633,840, 28.6.27. Appl., 24.9.24).—Maize starch is heated under pressure with dilute hydrochloric acid, the product being neutralised with sodium carbonate and mixed with finely-ground sodium hydroxide and granulated borax. T. S. WHEELER.

Treatment of leather. M. C. LAMB, Assr. to ROHM & HAAS CO., INC. (U.S.P. 1,640,706, 30.8.27. Appl., 6.3.26).—See E.P. 255,555; B., 1926, 839.

XVI.—AGRICULTURE.

Correlation of nutritive value with dry matter content of pastures. E. J. SHEEHY (Sci. Proc. Roy. Dublin Soc., 1927, **18**, 389—398).—The composition and nutritive value of two pastures of differing reputation were studied by removing yard square turves of each and replanting them together with the successive layers of their accompanying soil and subsoil in adjacent pits. Similar plots were also laid down in which both turves were broken up and sown with the same seed mixture. A third plot was afterwards included, obtained from the locality in which the other turves were laid down. The herbage of the good pasture contained a higher percentage of dry matter than that from the poorer pasture. Although there were great differences in botanical composition, the chemical composition of the dry matter differed but slightly. The digestibilities, determined with guinea pigs, were similar. The dry matter content of the herbage may be correlated with botanical composition. G. W. ROBINSON.

Soluble silicate content of soils. W. R. G. ATKINS (Sci. Proc. Roy. Dublin Soc., 1927, **18**, 433—436).—Results are obtained for soluble silica in aqueous extracts of soils, using a colorimetric molybdate method, details of which are given. The amounts obtained vary from 18 to 124 pts./million. The results are too few in number for any general conclusions to be drawn. G. W. ROBINSON.

Manuring trials with town sewage. W. ZIELSTORFF and A. KELLER (Z. Pflanz. Düng., 1927, **B 6**, 350—365).—Irrigation with sewage water containing the effluent from cellulose factories, which, as previously reported (B., 1926, 764), caused injury to crops, showed no after-effects in the second season. Town effluent containing little or no cellulose effluent improved the yield of mustard and oats. Effluents harmful to other crops did not injure grass land, but, on the other hand, did not increase the yield as did the ordinary sewage effluent alone. Oats treated with a mixed effluent adjusted to contain 75 g./litre of residue on evaporation showed a decreased yield of grain and a somewhat smaller amount of straw. A. G. POLLARD.

[Plant] stimulants. E. HASELHOFF and W. ELBERT (Landw. Versuchs-Stat., 1927, **106**, 285—322; cf. *ibid.*, 1922, **100**, 59).—An exhaustive series of experiments on the effect upon subsequent growth and yield of treating seeds of many different plants with solutions of manganese and magnesium salts and of certain proprietary materials leads to the conclusion that no case can be made out for any general stimulating action of such solutions upon plant growth. The results obtained by Popoff could not be confirmed. C. T. GIMINGHAM.

Practical value of soil experiments. HELLMERS (Z. Pflanz. Düng., 1927, **B 6**, 366—373).—The necessity of more than one type of experiment in the characterisation of a soil and its fertiliser needs is pointed out. In particular, the value of a study of soil and subsoil maps in conjunction with the determination of fertiliser requirement (Neubauer) and of the buffer capacity is emphasised. A. G. POLLARD.

Ammonium sulphate and soil acidity. G. HAGEN (Z. Pflanz. Düng., 1927, **B 6**, 337—349).—Evidence is

advanced that the continued use of ammonium sulphate tends to increase soil acidity. The acid condition of many German soils is, however, not attributable to the excessive use of ammonium sulphate, nor can its ordinary use with other fertilisers be counted a source of danger to crops, except on soils already acid. The commonly accepted relative fertiliser values of ammonium sulphate and sodium nitrate, viz., 94 : 100, depend on trials in which soil reaction has not been considered. Under comparable conditions in this respect, the ratio should be more favourable to ammonium sulphate.

A. G. POLLARD.

Granulated cyanamide and dicyanodiamide. G. LEFORT DES YLOUSES (Chim. et Ind., 1927, **18**, 216).—Conclusions adverse to "granulated cyanamide" based on cultures in pots are criticised as unfair. Dicyanodiamide which is present in this particular fertiliser is not directly poisonous to plants, but in certain proportions may paralyse the nitrifying bacteria. General experience has shown that the altered granules containing a large proportion of dicyanodiamide are as efficacious as a source of nitrogen as other forms of cyanamide, and cases are quoted in which cyanamide containing 80% of dicyanodiamide gave a better yield with potatoes than nitrate of lime. The explanation is that the hard granules break down too slowly for a poisonous concentration of dicyanodiamide to be reached on the soil.

C. IRWIN.

Determination of ammonia in fertilisers and soils by a cold method. A. DEMOLON (Ann. Falsif., 1927, **20**, 412—413).—A rapid current of air, freed from ammonia by sulphuric acid, is passed for 2½ hrs. through the material (0·5—2 g. of fertiliser or 25 g. of soil) mixed with a solution of sodium carbonate or soap lye, with the addition of alcohol if frothing occurs, and thence into standard sulphuric acid. The ammonia is determined by titrating the excess of sulphuric acid. F. R. ENNOS.

PATENTS.

Preserving seeds from attack by pests. SACCHARIN-FABR. A.-G., and A. KLAGES (E.P. 274,974, 1.5.26).—The seed is treated with a complex mercury salt of an aromatic base, or with a mixture of salts which, by interaction with water, produces the complex mercury salt. For example, 2 pts. of mercury aniline hydrochloride are finely ground with 1 pt. of sodium chloride, the mixture being made up by addition of sodium sulphate and a dyestuff to contain a desired amount of mercury. The product is readily soluble in water. C. T. GIMINGHAM.

Treatment of lime-nitrogen [calcium cyanamide]. J. M. A. STILLESEN (U.S.P. 1,633,200, 21.6.27. Appl., 8.8.19. Renewed 9.11.26).—Crude calcium cyanamide is treated with water and carbon dioxide under pressure at 135° to form carbamide and calcium carbonate.

T. S. WHEELER.

Treatment of phosphate rock. H. BLUMENBERG, JUN., Assr. to STOCKHOLDERS SYNDICATE (U.S.P. 1,638,677, 9.8.27. Appl., 23.6.25).—Ground phosphate rock is mixed with ammonium sulphate and heated in a closed retort at below 300°, to form ammonium phosphates and calcium sulphate. H. ROYAL-DAWSON.

Decomposition of crude phosphates. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assees. of F. RÜSBERG (G.P. 440,001, 22.6.23).—Dry, non-caking phosphatic fertilisers are produced directly by treating crude phosphates with a mixture of nitric acid of, *e.g.*, 50% strength and concentrated sulphuric acid in the presence of more than one equivalent of an alkali or ammonium sulphate, calculated on the weight of calcium nitrate formed during the decomposition. L. A. COLES.

Conversion of fertilisers (U.S.P. 1,632,312).—See XVIII.

XVII.—SUGARS; STARCHES; GUMS.

PATENTS.

Preservation of cane juice. A. K. BALLS (U.S.P. 1,634,348, 5.7.27. Appl., 30.7.26.).—Cane juice for subsequent use in growing yeast is inoculated with lactic acid bacilli, and kept at 55° until formation of acid ceases. T. S. WHEELER.

Production of glucose [dextrose] from materials containing cellulose. L. MEILER and H. SCHOLLER (U.S.P. 1,641,771, 6.9.27. Appl., 10.9.24. Conv., 26.3.21). —See G.P. 407,412; B., 1925, 518.

Recovery of hydrochloric acid in glucose manufacture (U.S.P. 1,633,877).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Constitution and determination of pectins and gums in grape wines and musts. L. SEMICHON and M. FLANZY (Ann. Falsif., 1927, **20**, 395—399).—The pectins of wines and musts consist of a methyl ester of pectic acid combined with glycerophosphates of calcium, magnesium, and a little aluminium. The proportion of glycerophosphate is practically constant in the freshly precipitated pectins. For the determination of pectins and gums, 100 c.c. of the wine or must, acidified with 1 c.c. of hydrochloric acid, are treated with alcohol so that the mixture contains 80% of alcohol by vol. After being kept for about 24 hrs., the precipitate consisting of the pectins and gums is collected, washed with 80% alcohol till free from acid, dried at 90°, and weighed. It is redissolved in cold water, 20 c.c. of 0·1*N*-potassium hydroxide free from carbonate are added, and the whole is heated under a reflux for 1 hr., cooled, and acidified with acetic acid. After adding 10% calcium chloride solution, the whole is again refluxed for ¼ hr., the precipitate of calcium pectate collected, washed with water, dried at 90—95°, and weighed. The calcium content of the precipitate has been found practically constant in all determinations, viz., 6·46%, giving a minimum mol. wt. for pectic acid of 581. The gums are precipitated from the filtrate by means of alcohol, filtered, washed with alcohol, dried at 90—95°, and weighed. The proportion of pectin may be obtained by difference, or may be expressed by the weight of calcium pectate obtained. F. R. ENNOS.

PATENTS.

Manufacture and propagation of yeast. DISTILLERS CO., LTD., and E. A. MEYER (E.P. 275,328—9, [A, B] 4.5.26).—(A) During the so-called continuous fermentation process, degeneration of the yeast is prevented by a frequent change of feeding material which

renders possible the control at different stages of the fermentation of the ratio between proteins and carbohydrates and the content of vitamins. (B) The medium in which the yeast propagation takes place is sterilised by the addition of a halogen, preferably chlorine, or of materials capable of liberating a halogen.

C. RANKEN.

Stopping or arresting alcoholic fermentation. H. BOULARD (F.P. 615,014, 23.4.26).—Fermenting liquor is heated to a point above the lethal, but below the usual sterilising temperature, *e.g.*, to 43°, the process being repeated if necessary. L. A. COLES.

Ageing of alcoholic liquors etc. SIEMENS & HALSKE, A.-G., H. ENGELHARDT, and K. ENGELHARDT (E.P. 275,454, 16.11.26. Conv., 21.10.26).—Alcoholic beverages and fruit juices are matured by the addition of ozone and the products of oxidation affecting the flavour removed by the addition to the ozonised liquid of powdered substances of density not exceeding 4, *e.g.*, burnt magnesia. C. RANKEN.

Conversion of chemical fertilisers to yeast and other organic substances. F. C. RAETH (U.S.P. 1,632,312, 14.6.27. Appl., 25.1.24).—A nutrient solution suitable for growing yeast contains dextrose, ammonium and magnesium sulphates, ammonium phosphate, potassium dihydrogen phosphate, sodium chloride, calcium carbonate, and traces of ferrous and manganese sulphates. Continued aeration of the solution is necessary. T. S. WHEELER.

Recovery of hydrochloric acid [in the manufacture of dextrose from wood waste]. H. TERRISSE and M. LÉVY (U.S.P. 1,633,877, 28.6.27. Appl., 15.3.21. Conv., 25.3.20; cf. E.P. 154,170; B., 1921, 484 A).—The mixture of lignin, dextrin, dextrose, and hydrochloric acid, obtained in the preparation of dextrose from wood, is introduced at the top of a cylindrical vessel containing a number of superposed hollow shelves, the passage of the material through the apparatus being directed by rotating rakes. The shelves are maintained at a temperature of 50°, and a current of air heated at the same temperature is passed through the cylinder in counter-current to the material. Vaporised hydrochloric acid is condensed from the air current, which is then returned to the cylinder. T. S. WHEELER.

Preservation of cane juice (U.S.P. 1,634,348).—See XVII.

Malted milk (U.S.P. 1,636,563).—See XIX.

XIX.—FOODS.

Determination of the freshness of milk. G. INICHOFF (Z. Unters. Lebensm., 1927, **53**, 435—449).—Morres' method of judging the freshness of milk has been critically examined. The method consists in titrating the acidity of 40 c.c. of milk with 0·1*N*-caustic soda and the "degree of freshness" by adding to an equal volume of milk the exact volume of 0·1*N*-sulphuric acid to cause the casein to separate. The sum of the two titers is called the "coagulation value," and should not be less than 26. Observations on a large number of samples over a period of two years showed variations within wide limits. Using samples of 100 c.c. each in the place of the 40 c.c., "coagulation values" between 56·8 and 95·0 were obtained. Values below 50·0 were rare, 22% of the tests being above 70·0 and 92% between 50 and 80. The fixing of a definite value for normal milk is therefore difficult, but any milk giving a value below 50 should be regarded with suspicion. Addition of water to milk lowers both its acidity and "freshness," the "coagulation value" being correspondingly reduced. Preservatives in most cases appreciably increase the "coagulation value," sodium fluoride being an exception, whilst salicylic and benzoic acids have no influence. Heating or freezing milk produces a lowering of both acidity and "freshness." In view of the wide variations amongst different classes of milk and the powerful influence of a number of factors, the method is not considered suitable for the examination of fresh milk. H. J. DOWDEN.

Significance of solids-not-fat for the detection of watered milk. A GRONOVER and F. TÜRK (Z. Unters. Lebensm., 1927, **53**, 520—524).—Teichert's observation that milk containing less than 8% of solids-not-fat must have been diluted with water is only true within certain limits. Amongst 650 samples collected at the time of milking, 14% contained 7—8·25% of solids-not-fat. It is shown that determination of the lowering of f.p. is a much more reliable means of detecting the addition of water, especially as in many cases the ordinary determinations of total solids, fat content, etc. give no indication of the addition. Measurement of the refractive index in such cases affords additional evidence.

H. J. DOWDEN.

Determination of chlorine in milk. F. MACH and W. LEPPER (Z. Unters. Lebensm., 1927, **53**, 454—458).—Filippo and Adriani's observation (cf. B., 1926, 847) that the ashing process leads to a loss of chlorine has been confirmed. Direct titration by Drost's method (cf. B., 1923, 994 A; 1925, 822) involves errors owing to the bulky precipitate, and the end-point of the Volhard titration is not sharp. The relative merits of several methods for the removal of proteins before titrating the chloride have been investigated. To 50 c.c. of milk, diluted to 400 c.c., 50 c.c. of nitric acid were added, the mixture being filtered after shaking. Determination of chlorine by the Volhard method in 100 c.c. of the clear filtrate gave a moderately sharp end-point in the presence of ether. Weiss' method, using as precipitant 25 c.c. of 20% aluminium sulphate solution and 20 c.c. of 8% caustic soda solution, gave a granular precipitate and a sharp end-point without the addition of ether. Satisfactory results were obtained by using tannic acid; 50 c.c. of milk diluted to 400 c.c. are shaken in succession with 5 c.c. of 10% tannic acid, 10 c.c. of 10% oxidised ferrous sulphate solution, saturated sodium carbonate solution to alkalinity, 1 c.c. of 3% hydrogen peroxide, and acetic acid in excess. The filtrate was clear and the end-point very sharp. The three methods gave concordant results, which were higher than those obtained by ashing processes. Application of the methods to milk powders gave similar results. Subsequent investigations showed that phosphotungstic acid is the simplest precipitant. To 50 c.c. of milk, diluted to about 400 c.c., are added 40 c.c. of phosphotungstic acid solution (50 g. with 500 c.c. of nitric acid, *d* 1·4, diluted to 1 litre). After shaking and filtering, 100 c.c. of the filtrate are

titrated by the Volhard method in the presence of ether. H. J. DOWDEN.

Simplified chlorine determination in milk. G. SCHULZE (Milch. Zentr., 1927, **56**, 157—160).—A method based on that of Drost (cf. B., 1923, 994 A), has been developed, which makes use of the so-called Martius-Lüttke's 0·1*N*-silver nitrate solution, consisting of 17·5 g. of silver nitrate, 900 c.c. of nitric acid diluted to 1 litre, and containing an iron indicator. 10 c.c. of milk are vigorously shaken with 5 c.c. of the silver nitrate solution, the protein and silver chloride being precipitated in fine particles. The excess silver is titrated with 0·1*N*-ammonium thiocyanate solution, with vigorous shaking after each addition. The end-point is best detected if the precipitate is allowed to settle. The results are in fair agreement with those obtained by the ashing process. H. J. DOWDEN.

Securing true average samples of milk. K. WEBER (Z. Unters. Lebensm., 1927, **53**, 449—454).—The fat contents have been determined in numerous samples of milk withdrawn from the vessel before and after stirring, by dipping the ladle to the bottom of the can, and after pouring several times from one vessel to another. The results show that a thorough stirring is sufficient for sampling purposes. H. J. DOWDEN.

Methylene Blue (reductase test) in milk grading. M. GRIMES, H. S. B. BARRETT, and J. REILLY (Sci. Proc. Roy. Dublin Soc., 1927, **18**, 437—441).—The decolorisation of Methylene Blue by reductase from bacteria has been proposed as the basis of a test for grading milk. It was found that samples of Methylene Blue of different origin were all suitable for the test, and that stock solutions were stable over a period of nine months. Variations between duplicates and triplicates were increased with longer reduction times. When the reduction time was less than 2 hrs., variations in the ratio of Methylene Blue to milk did not alter the result obtained.

G. W. ROBINSON.

Simplified molecular constant applied to milks from the Somme. G. JORET and E. RADET (Ann. Falsif., 1927, **20**, 341—353, 403—411).—Analyses are given of genuine milks from various herds fed in different parts of the Somme valley, together with their simplified molecular constants (C.M.S.). For 196 milks from individual cows the C.M.S. varies between 69·2 and 82·8, with an average value of 73·9. Only 5 fall below 70, which figure, with a margin of one unit below, is thus confirmed as the lower limit for genuine milks. Mixtures of milks from different cows often show lower C.M.S. values than those calculated from their constituent milks. The C.M.S. is influenced by the nature of the food, replacement of pasturage by oil cake or bran food tending to lower it, by the amount of food, the age of the cow, the stage of lactation, and the hour of milking.

F. R. ENNOS.

Testing the refractive index and lactose content of milk from individual herds. G. SCHULZE (Z. Unters. Lebensm., 1927, **53**, 509—520).—The variations in the composition of the milk from 5 herds of cows have been observed at regular intervals over a period of one year, determinations being made of the density, fat content, solids-not-fat, lactose, chlorine, and refractive index. In March and April, when grazing commenced, there was a perceptible fall in the lactose content of the milk, which was restored in winter when artificial feeding was resumed. The milk from cows which had been artificially fed throughout the year did not show any great variation in lactose content. Graphical representation of the results showed a distinct parallelism between the lactose content and the refraction of the calcium chloride serum. H. J. DOWDEN.

Determination of small amounts of benzoic acid in milk, butter, margarine, meat, and eggs. J. GROSSFELD (Z. Unters. Lebensm., 1927, **53**, 467—483).—The determination of benzoic acid involves three processes, viz., procuring an aqueous solution of the acid, extracting this solution by means of a solvent, determination of the extracted material. To secure a satisfactory aqueous solution avoidance of emulsions by removal of proteins is essential, the most suitable methods being steam distillation or precipitation with 15% solution of potassium ferrocyanide and 30% solution of zinc sulphate. For the extraction from the aqueous solution, benzene was found to be a satisfactory solvent. When, however, the concentration of benzoic acid is less than 5 mg./100 c.c., the partition coefficient is not constant and Berthelot's law no longer applicable. By shaking equal volumes of benzene and an aqueous solution of sodium benzoate acidified with 2 c.c. of sulphuric acid per 100 c.c. of benzoate solution, 80% of the benzoic acid is extracted by the benzene. For the determination of the extracted acid, weighing or titration with alkali are only applicable for relatively large quantities of material. For very low concentrations the colorimetric methods of Jones (cf. B., 1925, 824) are preferable. The red coloration produced by the Mohler reaction as modified by Grossfeld (cf. A., 1916, ii, 158) is compared with a standard iron solution mixed with thiocyanate solution. The details of procedure with the various foodstuffs are given. H. J. DOWDEN.

Microscopical determination of husk content of cocoa powders. R. TURNAU (Z. Unters. Lebensm., 1927, **53**, 483—486).—The number of scleridæ in pure cocoa powders, cacao husks, and commercial cocoas have been determined by a modification of Griebel and Sountag's method (cf. B., 1926, 605). The dry, fat-free substance (0·5 g.) in 10 c.c. of 0·1*N*-caustic soda and 90 c.c. of water is gently boiled for 1 hr. with 2·5 c.c. of 30% hydrogen peroxide. After cooling, a further 10 c.c. of 0·1*N*-caustic soda are added and the mixture is again boiled for ½ hr. After allowing the sediment to settle the staining and counting are performed as described by Griebel and Sonntag (*loc. cit.*). The cacao husks were ground to pass 0·15-mm. mesh, the coarse residue being dried for 2 hrs., reground, and sieved. The powdered material was then defatted by extraction with ether for 6 hrs., followed by refluxing with trichloroethylene (100 c.c. per 10 g. of material). The results were in good agreement with those obtained by Griebel and Sonntag, but appreciably lower than those of Plücker (cf. B., 1926, 105). One of the commercial powders showed a number of scleridæ greater than 400 per mg. of fat-free substance, which Griebel and Sonntag regarded as the highest permissible number for unadulterated cocoa. H. J. DOWDEN.

Analysis of conserves containing apple and fruit (determination of the apple–fruit ratio). C. F. Muttelet (Ann. Falsif., 1927, **20**, 391—394).—The citric acid content of fruit juices used in mixed fruit and apple conserves is practically constant for each type of fruit, the minimum value for black currants being 3%, for gooseberries 2%, and for raspberries and strawberries 1%. Apple juice contains no citric acid. In the absence of added citric acid, the fruit juice content in a conserve may be calculated from the citric acid contents of the conserve and of the fruit juice. The apple content is obtained by deducting that of the sugar and of the fruit juice from 100. F. R. Ennos.

"Mikrobin" [sodium *p*-chlorobenzoate as food preservative]. C. von der Heide and R. Föllen (Z. Unters. Lebensm., 1927, **53**, 487—509).—Methods for the detection and determination of *p*-chlorobenzoic acid, its solubility in water, alcohol, and fruit juices, and its fungicidal efficiency have been investigated. Mohler's reaction as modified by von der Heide and Jakob (cf. A., 1910, ii, 359) is used to distinguish benzoic acid from the *p*-chloro-acid. The alkaline extract is evaporated to dryness and nitrated at 130—140° with a few drops of sulphuric acid and nitric acid. After cooling, excess of ammonia solution is added, followed by 10% solution of sodium sulphide until a reddish-brown colour appears. With *p*-chlorobenzoic acid the colour deepens on boiling, but with benzoic acid it vanishes. With hydroxylamine, nitrated benzoic acid gives a red coloration, *p*-chlorobenzoic acid no colour. For the determination of *p*-chlorobenzoic acid in foods, the acid is separated by steam distillation or by ether extraction, and chlorine determined in the distillate or extract by evaporating to dryness, treating with sodium peroxide, gently igniting, and precipitating as silver chloride. Ether extraction gives slightly low results, and is recommended for food analysis. The solubility of *p*-chlorobenzoic acid in water–alcohol mixtures varies from 0·078 g./litre for water to 31·3 g./litre for pure alcohol. The relative efficiencies of benzoic, salicylic, and *p*-chlorobenzoic acids in arresting the growth of micro-organisms have been investigated. Against yeast fermentation *p*-chlorobenzoic acid was most effective, against moulds benzoic acid, whilst against mother of vinegar benzoic and salicylic acids were both more potent than *p*-chlorobenzoic acid, but the tests in this case were not under anaerobic conditions. The statement of Neissen and Tillmans that mikrobin may be used in foodstuffs, provided it is declared, is strongly opposed, since the addition to wines of mikrobin is already prohibited. H. J. Dowden.

Nutritive value of pastures. Sheehy.—See XVI.

Determination of water. Cantzler and Rothschild.—See XX.

Patents.

Manufacture of yeast foam malted milk. C. B. Hill and M. H. Givens, Assrs. to Northwestern Yeast Co. (U.S.P. 1,636,563, 19.7.27. Appl., 12.3.26. Cf. U.S.P. 1,589,853; B., 1926, 719).—Yeast is grown on a nutritive medium of cereal flour treated with a malt cereal, after which the mixture is mashed, neutralised if necessary, and heated at 88—97° for 30—60 min. to stop yeast growth and fermentation, and also to gelatinise the starch, dissipate alcohol formed, and sterilise the product. Milk is then added, the whole heated at 70° for at least 30 min, and dried to a powder if desired. F. R. Ennos.

Manufacture of processed cheese. E. E. Eldredge, Assr. to Pabst Corp. (U.S.P. 1,634,410, 5.7.27. Appl., 19.7.24).—Neutralised whey is concentrated to a paste and mixed with ground cheese (9 pts.), the mixture being then pasteurised. T. S. Wheeler.

[Protectively] treating food products. W. W. Willison, Assr. to Thermokept Corp. (U.S.P. 1,637,068, 26.7.27. Appl., 2.2.24).—The selected food product, while subjected to a high vacuum, is completely immersed in a suitable liquid, *e.g.*, water or brine, below 38°, and the vacuum maintained until the occluded gases are removed and the mass becomes quiescent. The vacuum is then broken, thus allowing the liquid to penetrate and surround the cell structure of the food product, and the whole is heated at 100° to hydrolyse the starch, which then acts as a protective seal. F. R. Ennos.

Extraction of volatile odorous constituents of oils, especially edible oils. N. C. Furbo (G.P. 439,615, 14.5.24).—The oil is treated with a countercurrent of gas or steam in apparatus comprising a number of superposed, rotating, concave plates. The oil is fed on to each plate near the centre, and flows over it in a thin film towards the outer lip, whence it is conveyed to the next plate. L. A. Coles.

Manufacture of edible substances. P. N. Woo (E.P. 269,576, 14.4.27. Conv., 15.4.26).—See U.S.P. 1,602,958; B., 1927, 26.

Beating of viscid liquids [*e.g.*, eggs] (E.P. 274,976).—See I.

Active charcoal from cacao refuse (G.P. 440,381).—See II.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of water in organic substances by means of calcium carbide. A. Cantzler and S. Rothschild (Z. Unters. Lebensm., 1927, **53**, 425—435).—About 3 g. of the substance are weighed in a flask in which is a small beaker containing about 5 g. of calcium carbide. The flask is fitted with inlet and outlet tubes which are closed during the weighing. The inlet tube is then connected to a series of soda–lime and drying tubes, the outlet tube being fitted to an aspirating device. After overturning the beaker and mixing the calcium carbide with the substance under examination, the flask is heated for 1 hr. on a toluene bath at 60—100° according to the nature of the material. Residual acetylene is removed by a current of dry air free from carbon dioxide, and, after cooling, the loss of weight is determined. Theoretically, 1 g. of acetylene is equivalent to 1·385 g. of water, but blank experiments using sand moistened with a known weight of water showed that this factor is too low, and that 1·443 gives more accurate results, the disparity being attributed to the fact that part of the water is absorbed by the lime formed. The results with the higher factor are in good agreement with those obtained by vacuum desicca-

tion. For such materials as spices and seasoned foodstuffs the method has the advantage that there is no risk of volatilising the ethereal oils. The use of aluminium carbide in place of calcium carbide and determination of the methane evolved was found to be unsatisfactory. H. J. DOWDEN.

Determination of total citric acid in solution of magnesium citrate. J. L. MAYER (J. Amer. Pharm. Assoc., 1927, **16**, 719—722).—The method given in the U.S. Pharmacopœia X for the examination of "Solution of magnesium citrate" is criticised, and a modification is described in which the titration of free citric acid is carried out in a platinum dish, the residual solution being evaporated and ashed prior to titrating for combined citric acid. Magnesium may be determined gravimetrically in the final residual solution, but it is claimed that determinations made directly on a solution of the citrate are just as accurate. C. O. HARVEY.

Determination of nicotine in tobacco. L. FRANK (Chem.-Ztg., 1927, **51**, 658).—10 g. of finely-divided air-dried tobacco are shaken for ½ hr. with 10 c.c. of 20% potassium hydroxide solution, 100 c.c. of ether, and 100 c.c. of light petroleum in a graduated Röhrig separator having a side tube and tap. After allowing to settle for several hours, 100 c.c. of the clear liquid are drawn off, filtered through ignited sodium sulphate, the filter washed with dry ether, and the solvent distilled from the filtrate. The greenish residue is extracted with cold water, collected on a filter, and the colourless filtrate titrated with 0·1*N*-hydrochloric acid, using Congo Red as indicator. Test results were in satisfactory agreement with those obtained by the standard methods. F. R. ENNOS.

Solubilities of drugs in glycerol. J. A. ROBORGH (Diss., Amsterdam, 1927, 1—45).—The solubilities in 86·5% and 98·2% glycerol of the following substances have been determined: ethyl acetate, stearic acid, anæsthesine, sodium arsenate, ammonium carbonate, guaiacol, guaiacol carbonate, codeine hydrochloride, calcium and sodium hypophosphites, novocaine, phenacetin, phenol, sodium pyrophosphate, atropine sulphate, tannin, quinine tannate, theobromine, zinc valerate, vanillin. CHEMICAL ABSTRACTS.

Theoretical structure of the correction factor as applied in the menthol assay of peppermint oil. Assay of oil of rosemary. S. MENDELSOHN (J. Amer. Pharm. Assoc., 1927, **16**, 726—729).—The method of calculation of the menthol content of peppermint oil from figures obtained by saponification of the acetylated oil as given in the U.S. Pharmacopœia X is explained. Modification of Power and Kleber's original formula (cf. B., 1895, 594) is necessary owing to the presence of up to 10% of menthol esters in the oil. The same correction factor is applied in the assay of oil of rosemary, though in this case the figure sought is the % of total borneol. The official method assumes that menthol and borneol are the only acetylatable constituents, and that they are the only alcohols and their acetates the only esters present. As none of these assumptions is strictly correct, Parry's method (involving a determination of combined alcohols by saponification and of total alcohols in the saponified oil) is recommended, the necessary correction for the reduction in weight during saponification being derived from the amount of alkali absorbed. C. O. HARVEY.

Determination of benzoic acid. GROSSFELD. **"Mikrobin" as preservative.** VON DER HEIDE and FÖLLEN.—See XIX.

PATENTS.

Manufacture of formates. L'AIR LIQUIDE, SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (E.P. 246,168, 16.1.26. Conv., 17.1.25).—Formates are prepared by the action of carbon monoxide or gases containing it (especially gases rich in it separated by partial liquefaction of water-gas etc. containing it together with hydrogen) on caustic alkali solutions or on suspensions of alkaline-earth hydroxides in liquids or in alkali carbonate solutions, at pressures above 300 atm. and at a temperature about 400°. By the use of concentrated solutions, *e.g.*, those containing 400 g. of sodium hydroxide per litre, solutions of formates containing 700 g. per litre are formed. B. FULLMAN.

Production of acetaldehyde. A. E. CRAVER, Assr. to BARRETT Co. (U.S.P. 1,636,952, 26.7.27. Appl., 13.3.22).—A mixture of ethyl alcohol and air (14 pts.) is passed at about 300° over a catalyst comprising vanadium oxide, with or without addition of molybdenum oxide, the time of contact being about 0·4 sec. T. S. WHEELER.

Manufacture of alkali xanthates. W. HIRSCHKIND, Assr. to GREAT WESTERN ELECTRO CHEMICAL Co. (U.S.P. 1,636,229, 19.7.27. Appl., 3.11.25; cf. U.S.P. 1,507,089; B., 1924, 908).—The addition of a small quantity of a chlorohydrocarbon, *e.g.*, carbon tetrachloride, to the mixture of carbon disulphide, alcohol, and alkali hydroxide employed in the preparation of alkali xanthates, enables the reaction to be carried out at a lower temperature (10°) with increased yield. T. S. WHEELER.

Production of maleic acid. A. E. CRAVER, Assr. to BARRETT Co. (U.S.P. 1,636,857, 26.7.27. Appl., 23.5.23).—A mixture of benzene vapour and air (13 pts.) is passed at about 500° over a catalyst containing vanadium oxide (70%) and molybdenum oxide (30%), the time of contact being about 0·3 sec. T. S. WHEELER.

Manufacture of lactic acid esters. CANADIAN ELECTRO PRODUCTS Co., LTD., Assees. of H. W. MATHESON and K. G. BLAIKIE (E.P. 257,907, 11.8.26. Conv., 3.9.25).—Acetaldehydecyanohydrin (lactonitrile) is treated with aqueous alcoholic hydrogen chloride containing not more than 1 mol. of water and a small excess of hydrogen chloride. Ethyl lactate, b.p. 148—150°, is obtained in 90% yield by passing hydrogen chloride into a mixture of ethyl alcohol, lactonitrile, and 35·5% hydrochloric acid at 30—35° until 1·01 mol. of hydrogen chloride is present, and heating for 3 hrs. at 80°. Alcoholic hydrogen chloride may be used instead of the gas. C. HOLLINS.

Manufacture of esters of α-hydroxy-acids. CANADIAN ELECTRO PRODUCTS Co., LTD., Assees. of H. W. MATHESON and K. G. BLAIKIE (E.P. 264,143, 18.12.26. Conv., 11.1.26. Cf. E.P. 257,907; preceding abstract).—

The process of the prior patent is applied generally to the conversion of cyanohydrins of aldehydes and ketones (excluding acetaldehyde) into esters of α-hydroxy-acids. Examples are ethyl furylglycollate, ethyl α-hydroxy-*n*-valerate, ethyl α-hydroxy*iso*butyrate, and ethyl mandelate. C. Hollins.

Manufacture of hexamethylenetetramine. C. B. Carter, Assr. to S. Karpen & Bros. (U.S.P. 1,635,707, 12.7.27. Appl., 9.2.24; cf. U.S.P. 1,566,820—2; B., 1926, 217).—Methylene chloride is heated at 100° under pressure with a large excess of ammonia solution saturated with hexamethylenetatramine and ammonium chloride. The resulting liquor is cooled, treated with ammonia, and used again, the mixture of hexamethylenetetramine and ammonium chloride precipitating being separated into its components in any suitable manner. T. S. Wheeler.

Manufacture of furan. W. C. Wilson, Assr. to Quaker Oats Co. (U.S.P. 1,636,030, 19.7.27. Appl., 28.11.24).—Furfuroic acid if heated at 250° partly sublimes and the remainder decomposes to furan and carbon dioxide. The acid is separated by cooling the vapours to 40°, carbon dioxide being removed with sodium hydroxide solution, and furan condensed by cooling with brine. T. S. Wheeler.

Barbituric acid derivative. E. H. Volwiler, Assr. to Abbott Laboratories (U.S.P. 1,636,201, 19.7.27. Appl., 16.7.26).—5-*n*-Butylbarbituric acid is treated with allyl bromide in presence of concentrated sodium hydroxide solution in the cold, to give 5-n-*butyl-5-allylbarbituric acid*, m.p. 128°. T. S. Wheeler.

Manufacture of alkoxyamino-[8-amino-6-alkoxy-] quinolines. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 275,277, 4.2.26).—8-Nitro- or 8-azo derivatives of 6-alkoxyquinolines are reduced, or 8-amino-6-hydroxyquinoline is alkylated, or 8-acylamino- or 8-arylideneamino-6-hydroxyquinolines are alkylated and hydrolysed, the process of E.P. 267,457 (B., 1927, 379) being excluded. 8-Amino-6-methoxyquinoline, m.p. 41°, is obtained by reducing the nitro-compound with iron and acetic acid. 8-Amino-6-hydroxyquinoline, m.p. 177°, obtained from the 8-nitro-compound, m.p. 239—240° (decomp.), prepared by Skraup's reaction or by demethylation of the methyl ether, is converted into the formyl derivative, ethylated with sodium ethoxide and ethyl iodide, and hydrolysed to give 8-amino-6-ethoxyquinoline, m.p. 60°, b.p. 144—145°/1 mm. 8-Amino-6-hydroxyquinoline, treated with methyl sulphate and alkali, gives 8-amino-6-methoxyquinoline, b.p. 145—147°/2 mm. C. Hollins.

Manufacture of preparation which emits β-rays. M. Wreschner and L. F. Loeb (E.P. 265,600, 3.2.27. Conv., 8.2.26. Addn. to E.P. 248,765; B., 1926, 768).—The process of the prior patent is modified in that the ferric salt there used is replaced by an aluminium salt. B. Fullman.

Manufacture of betaine hydrochloride. D. K. Tressler, Assr. to Larrowe Construction Co. (U.S.P. 1,634,221, 28.6.27. Appl., 13.7.25).—The process described in E.P. 265,831 (B., 1927, 423) is modified in that in place of hydrochloric acid, a mixture of sulphuric acid and calcium chloride is added to the solution under treatment. T. S. Wheeler.

Manufacture of a glucoside of *Adonis Vernalis*, L. F. Hoffmann-La Roche & Co., A.-G. (E.P. 265,941, 20.1.27. Conv., 13.2.26).—The aqueous or alcoholic extract of *herba adonidis vernalis* is treated directly, or after treatment with lead acetate, with an adsorbing agent such as animal charcoal. The latter is extracted with chloroform, and the chloroform solution concentrated and poured into light petroleum. The cardioactive glucoside separates as a thick oil, which, on dissolution in water and evaporation *in vacuo*, yields a brown powder. B. Fullman.

Preparation of stable colloidal silver solutions. W. Traxl (U.S.P. 1,633,466, 21.6.27. Appl., 6.3.24. Conv., 13.3.23).—A mixture of silver oxide and hydroxide (1%) is added to an oil or fat at 250°, the solution obtained being filtered. The filtrate is treated with hydrogen, and the process is repeated several times. The product has therapeutic value. T. S. Wheeler.

Stable and sterilisable solutions containing organic phosphorus and complex auro-compounds. I. G. Farbenind. A.-G., Assees. of L. Benda (U.S.P. 1,635,169, 12.7.27. Appl., 18.6.26. Conv., 22.6.25).—A dialkylaminoarylphosphinous acid, *e.g.*, *p*-dimethylaminotolylphosphinous acid, is dissolved as the sodium salt in a sodium aurothiosulphate solution containing sodium thiosulphate, the resulting solution being sterilised with steam and employed in the treatment of tuberculosis. T. S. Wheeler.

Manufacture of synthetic camphor by means of a liquid catalyst. L. and E. Darrasse, and L. Dupont (U.S.P. 1,641,579, 6.9.27. Appl., 1.6.21. Conv., 9.6.20).—See E.P. 164,357; B., 1922, 610 a.

Manufacture of borneol esters. H. Blum, Assr. to Soc. Alsacienne de Prod. Chim. (U.S.P. 1,640,639, 30.8.27. Appl., 18.3.25. Conv., 28 3.24).—See E.P. 231,468; B., 1925, 900.

Manufacture of betaine hydrochloride. D. K. Tressler, Assr. to Larrowe Construction Co. (U.S.P. 1,634,221, 28.6.27. Appl., 2.7.24).—See E.P. 265,831; B., 1927, 423.

Manufacture of diarylguanidines. C. J. T. Cronshaw and W. J. S. Naunton, Assrs. to Brit. Dyestuffs Corp., Ltd. (U.S.P. 1,639,724—5, 23.8.27. Appl., [a] 28.6.26, [b] 2.3.27. Conv., [a, b] 4.7.25).—See E.P. 255,220; B., 1926, 769.

Manufacture of metallo-organic [alkyl] compounds. C. A. Kraus and C. C. Callis, Assrs. to Standard Development Co. (U.S.P. 1,639,947, 23.8.27. Appl., 13.4.23).—See E.P. 214,221; B., 1925, 827.

Heavy-metal mercaptosulphonic [acid] compounds. Chem. Fabr. auf Aktien (vorm. E. Schering), Assees. of A. Feldt, W. Schoeller, and E. Borgwardt (U.S.P. 1,633,626, 28.6.27. Appl., 10.12.25. Conv., 15.12.24).—See E.P. 266,824; B., 1927, 348.

Catalyst carrier (U.S.P. 1,636,685).—See I.

Hydroxy-compounds from amines (E.P. 274,960). **Catalytic methylation of ammonia and amines** (E.P. 275,377). **Catalytic oxidation of hydrocarbons** (E.P. 275,321).—See IV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Patents.

Production of coloured photographic films. L. F. Douglass (U.S.P. 1,632,278, 14.6.27. Appl., 16.10.19).—Prints of corresponding red and green colour selection negatives are produced on a positive film, comprising a transparent carrier sensitised on one side, by exposure from opposite sides, the outer red image being mordanted after development by treatment in a solution containing, amongst other ingredients, potassium dichromate, ferric alum, oxalic acid, and potassium ferricyanide. The image is dyed red and treated with alkali to remove any blue colour deposited on it. The green inner image is then treated by prolonged immersion in the mordanting bath, and the blue colour deposited on it is converted into green in a bath containing chromic acid and potassium ferricyanide.

T. S. Wheeler.

Manufacture of kinematograph colour films. J. E. Thornton (E.P. 275,331, 5.5.26).

XXII.—EXPLOSIVES; MATCHES.

[Continuous] nitroglycerin manufacture. A. Schmid (Z. Ges. Schiess- u. Sprengstoffw., 1927, 22, 169—173, 201—206).—It is proposed to nitrate glycerin continuously in a nitrator containing a series of water-cooled coils, the inner one having its turns so close together that it forms a partition between the centre of the nitrator, which contains a stirrer, and the remaining coils. The nitrator is filled with an emulsion of nitroglycerin and waste acid. Glycerin enters at the top of the nitrator and mixed acid at the bottom; the latter mixes with the emulsion and is forced by the action of the stirrer to the bottom of the nitrator and then up through the coils. Working details of the temperature and acid composition are given. The emulsion of nitroglycerin and spent acid flows from the nitrator into a separator, where it first passes through perforated plates, most of the nitroglycerin being thereby separated, and then over sloping corrugated plates from which nitroglycerin passes off at the top and the waste acid lower down. There is no after-separation. Washing takes place in an earthenware tower which contains perforated plates. It is claimed that this process reduces risk, decreases capital expenditure and maintenance charges, and gives an increased yield of nitroglycerin with smaller consumption of nitric acid.

S. Binning.

Recovery of solvents. Bodewig.—See I.

Patents.

Production of finely-crystalline lead or other heavy-metal azides. A.-G. Lignose (G.P. 440,568, 15.9.21).—Solutions of sodium azide and a soluble lead salt, *e.g.*, lead acetate, flow simultaneously into a funnel-shaped receiver from containers with the outlets regulated to deliver equivalent volumes of the two solutions.

L. A. Coles.

Manufacture of match-striking compositions. H. W. Robinson (U.S.P. 1,640,224, 23.8.27. Appl., 18.2.26. Conv., 4.2.26).—See E.P. 265,378; B., 1927, 269.

Manufacture of nitrated cellulose from wood pulp. V. Planchon (U.S.P. 1,641,292, 6.9.27. Appl., 27.2.25. Conv., 29.2.24).—See E.P. 230,092; B., 1925, 902.

XXIII.—SANITATION; WATER PURIFICATION.

Zeolite water treatment in a large central-heating plant. A. H. White, J. H. Walker, E. P. Partridge, and L. F. Collins (J. Amer. Water Works Assoc., 1927, 18, 219—249).—Preliminary experiments indicated that the high caustic alkalinity in boilers resulting from the use of a zeolite system with water high in bicarbonate could be safely reduced by partial neutralisation of the bicarbonate in the softened water by addition of sulphuric acid with subsequent removal of the carbon dioxide set free by the passage of steam. In the case of a large central-heating plant satisfactory results were obtained when about 50% of the bicarbonate in the zeolite-treated water was neutralised by sulphuric acid and the water then passed through a deaerating heater. After such treatment water had p_H 6·4 on entering the deaerator and 8·5 on leaving the deaerator and entering the boiler. Inspection of the boiler after eight months of operation revealed clean tubes and no serious corrosion. For more complete treatment an auxiliary feed of phosphoric acid instead of the addition of further quantities of sulphuric acid appears feasible, and would avoid excessive lowering of the p_H of the water.

W. T. Lockett.

Determination of sulphuric acid in water analysis by means of benzidine. L. W. Haase (Chem.-Ztg., 1927, 51, 637—638).—The precipitation of benzidine sulphate is not affected by the presence of salts of calcium, ferrous iron, or manganese, or by small quantities of chlorides, nitrites, or nitrates, provided the liquid is faintly acid. Magnesium and ferric iron tend to prevent the precipitation, but the effect of the latter may be counteracted by boiling with a few c.c. of 1% hydroxylamine hydrochloride. The determination of sulphate by precipitation of the benzidine salt and titration of the base liberated after hydrolysis gives results in good agreement with the gravimetric barium sulphate method for concentrations of SO_3 of 100 mg./litre and over; for smaller concentrations very low results are obtained, particularly when the SO_3 falls below 60 mg./litre. In order to render the method applicable to drinking water, and waters in which the SO_3 content is lower than 100 mg./litre, concentration to about one quarter bulk with addition of 5—10 c.c. of 1% hydroxylamine hydrochloride is necessary before precipitation.

F. R. Ennos.

Manuring trials with town sewage. Zielstorff and Keller.—See XVI.

Patents.

Purification of waste water. T. Franz (G.P. 430,669, 22.1.24).—Waste water containing clay or earthy impurities in suspension is mixed with water containing less than ½% of cutting oil, with or without the addition of small quantities of substances which by colloid-chemical or physical action can facilitate separation of the impurities.

L. A. Coles.

Purifying air etc. (E.P. 274,942).—See I.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCTOBER 28, 1927.

I.—GENERAL; PLANT; MACHINERY.

Estimation of the efficiency and dispersive power of emulsifying agents. R. C. Smith (J.S.C.I., 1927, **46,** 345—346 T).—The criteria of a good emulsifying agent are (1) for a given quantity the agent should emulsify as large a fraction of the disperse phase as possible; and (2) the disperse phase should be in as small globules as possible. Two methods are given for the numerical expression of the results. The "emulsifying power" is the fraction of the disperse phase emulsified, and the "dispersion power" is the fraction of the total volume occupied by the emulsion. To obtain one representative figure the product of these two is taken. This method breaks down when a very small quantity of disperse phase is dispersed throughout the whole of the dispersion medium, so a second method is suggested. If unity represents the volume of disperse phase before emulsifying, then the "dispersion factor" represents the total volume occupied by emulsion and remaining disperse phase. In all cases equal quantities of dispersion medium and disperse phase are taken. Tables are given showing typical results.

Inexpensive and accurate gas chain [hydrogen electrode vessel] for liquids lighter than saturated potassium chloride solution. H. C. Waterman (J. Assoc. Off. Agric. Chem., 1927, **10,** 390—395).—A simple type of sharp-junction hydrogen electrode vessel of the bubbling type is described, which requires only a small volume of the solution under examination and ensures the freedom of the latter from potassium chloride contamination and exclusion of carbon dioxide. The apparatus can also be adapted for use with foaming liquids or those containing sediments. F. R. Ennos.

Works viscosimeter. R. Freund (Farben-Ztg., 1927, **32,** 2889—2890).—A simplification of the "falling sphere" method of determination of viscosity is described. The sphere is attached to a suitably marked fine thread which is allowed to slip loosely through the fingers of the operator, the time taken for the sphere to fall through a given distance being observed. It is not necessary to transfer the sample of fluid under test to a special vessel, and the sphere may readily be recovered, both points of advantage over the usual method. The accuracy, although decreased, is sufficient for normal works practice. S. S. Woolf.

Flowmeter for gases. E. H. Riesenfeld (Chem.-Ztg., 1927, **51,** 678—680).—The apparatus consists of a manometer filled with a suitable liquid and provided with scales to each arm. The two arms are joined at the upper end with a horizontal capillary tube which may be sealed to the manometer, attached by rubber connexions, or provided with a cooling apparatus. The upper ends of the manometer tubes are provided with bulbs to prevent loss of liquid, and the tube joining the lower end of the tubes is of small bore to hinder rapid movement of the liquid. The diameter of the capillary tube is adjusted to allow of measurement of rates of flow as low as 0·1 litre/hr. Details of the method of standardising the scale and of the most suitable liquids for different purposes are given. A. R. Powell.

Tower absorption coefficients. R. C. Cantelo, C. W. Simmons, E. M. Giles, and F. A. Brill (Ind. Eng. Chem., 1927, **19,** 989—992).—The rate of dissolution of a soluble gas from a gas mixture in an absorbing liquid is given by $dm/dt = -K_2f(m)$, where m is the concentration of the gas in the gas mixture and K_2 the dissolution coefficient. This equation was further investigated for the cases of carbon dioxide and sulphur dioxide, a tower packed with Raschig rings being used. With carbon dioxide it was found that K_2 was scarcely affected by changes in the gas velocity or composition, the flow of water and temperature being constant. With increase in water flow K_2 decreased rapidly to a critical point and then remained constant. Solutions of sulphur dioxide at higher temperatures follow Henry's law closely, though there is deviation at lower temperatures. Under the conditions of the experiment K_2 increased linearly with the rate of flow of water. In each case the quantity K_2 is calculated from the quantity relationships of gas and solution, temperature, vapour pressure, Henry coefficient, etc. C. Irwin.

Patents.

Kilns. M. M. Minter (E.P. 275,858, 18.6.26).—An arrangement of flues by which the heat from a burning kiln may be used to preheat a neighbouring one and by which heated air from a cooling kiln can be delivered to the fire doors. B. M. Venables.

Retort. H. J. and H. C. McElvain (U.S.P. 1,642,457, 13.9.27. Appl., 12.6.26).—Concentric, spaced inner and outer rotary cylinders are mounted within an inclined stationary cylinder provided with peripheral intake and discharge openings. The innermost cylinder is the longest, and means are provided for supplying heat within it. The adjacent surfaces of the rotary cylinders carry longitudinal wings. H. Holmes.

Grinding and separating apparatus. P. L. Crowe (U.S.P. 1,639,214, 16.8.27. Appl., 20.10.25).—A grinding cylinder provided with apertures is journalled on a base, and a crusher plate is yieldingly urged toward it. Means are provided for producing a current of air through the apertures for withdrawing the finely-ground material. H. Holmes.

Mixing or pugging mill. C. J. Cooper and A. M. Mason (C. J. Cooper & Co.) (E.P. 276,196, 1.11.26).—The casing is of frusto-conical shape with axis horizontal and outlet at the wider end, but there are two cones, with incomplete circumferences, joined together, and two sets of beaters or scrapers the blades of which intercalate and overlap; provision is also made for scraping the ends of the casing. B. M. Venables.

Dryer. G. Komarek, Assr. to Malcolmson Engineering & Machine Corp. (U.S.P. 1,641,337, 6.9.27. Appl., 12.2.24).—The space between an inner rotary cylinder and an outer stationary tubular housing constitutes an annular drying chamber. Heating medium is supplied to one end of the cylinder through a heat-regulating chamber and detachable tubular connexions. H. Holmes.

Drying and heating apparatus. L. B. West (U.S.P. 1,641,108, 30.8.27. Appl., 19.3.26).—The material to be treated is delivered into the space between an outer dryer casing and an inner superheater, conveyed into the superheater, and discharged therefrom. One end of the superheater extends beyond the casing into a draught chamber communicating with the space between them, and hot gases are admitted from a flue to one end of the superheater. Means are provided for rotating the casing and the superheater. H. Holmes.

[Waterproofing of] tanks, vats, pits, etc. N. Swindin (E.P. 275,316, 3.5.26).—Rubberised cement of known composition (*e.g.*, Portland cement mixed with latex) is applied to the internal surface of the vessel to be waterproofed, and sheets of rubber are then secured to the cement by means of rubber solution. B. M. Venables.

Separation of materials by [vacuum] flotation. F. E. Elmore (E.P. 275,778, 16.7.26).—The pulp of mineral, water, and oil or other selective agent is drawn up by a vacuum through a vertical feed pipe in a non-turbulent stream, the velocity of the flow being kept constant by increasing the cross-section of the pipe in proportion to the increase in volume of pulp due to the vacuum. In the separating chamber the outlet of the feed pipe is surrounded by baffles forming louvres, and at about that level also are radial valves to prevent rotation and disturbance of the froth, but below that level, in the zone which ought to be occupied only by non-floatable mineral or gangue, the pulp is put into rotation by tangential, internal water jets in order that any stray particles of float may get clear and rise. The outlet for gangue is provided with a valve which is intermittently opened and produces pulsations which help to prevent channelling and clogging of the float. B. M. Venables.

Apparatus for the chemical and mechanical treatment of mixed liquids and solids. T. Steen (G.P. 440,800, 30.1.25).—Apparatus suitable for dressing heavy spar, for treating ores with acids, and for processes involving the double decomposition of salts comprises a closed vessel fitted with two pumps of the type used for elevating mixtures of gases and liquids (mammut pumps), one of which, with its base set lower in the vessel than the other, serves to mix the material and to regulate the pressure in the vessel as soon as the second pump, which removes the material from the vessel, comes into operation. L. A. Coles.

Settling apparatus [jigs] for washing granular materials. K. and E. Gröppel, A. Waschkau (Maschinenfabr. F. Gröppel, C. Luhrig's Nachf.), and W. Scholvien (E.P. 253,139, 4.6.26. Conv., 6.6.25).—In addition to the vertical motion given by the usual cranks or other means, the tray of a jig is given a horizontal motion by means of guides which may be straight inclined, curved partly inclined, or may follow a closed path so that on the upstroke the forward movement takes place after bedding of the material has taken place. B. M. Venables.

Manufacture of colloidally dispersed material. S. Cabot (E.P. 246,874, 1.2.26. Conv., 31.1.25).—A permanent colloidal suspension of solids is produced in non-solvent media by kneading the solid with a proportion of a gel substance so small that it is only sufficient to film-coat the solid particles, the materials being in the form of a stiff, plastic, and sticky paste. A small quantity of the dispersion medium may be added during the kneading if necessary to reduce the gel colloid to a soft condition. *E.g.*, 3 pts. by wt. of a titanium oxide-barium sulphate mixture ("Titanox") are kneaded with 1 pt. of linseed oil jelly containing 30% of turpentine, then to the paste are added 1 pt. of raw linseed oil and enough turpentine and drier to make a workable paint in the form of a permanent suspension. B. M. Venables.

Centrifugal separating apparatus. Brit. Separators, Ltd., and A. G. Cahill (E.P. 276,268, 7.4.27).—In a separator for liquids where it is desirable for the heavier liquid to carry off any solid matter, light flocculent matter will be carried off through the ordinary outlet passages starting near the circumference, but should these become choked by gummy or heavy solids an alternative route is provided for the heavier liquid starting further inwards, but not as far in as the boundary of the lighter liquid. B. M. Venables.

Evaporation of liquids. E. Morterud (E.P. 263,132, 10.12.26. Conv., 19.12.25).—A number of superposed shallow trays or troughs for the liquid are contained in a common vapour chamber, and are heated by steam tubes or other elements which extend vertically through all the trays. The trays are reciprocated vertically, and are provided with scrapers having a certain amount of freedom which surround the heating tubes and keep their exterior surfaces clean. B. M. Venables.

Recovering dissolved substances from solutions. Metallbank u. Metallurgische Ges. A.-G. (G.P. 440,237, 7.8.21).—The liquid is allowed to trickle down a series of heated, inclined surfaces in a tower, the length of the individual surfaces, the rate of heating, and the rate of flow of the liquid being so adjusted that the solid material forms a thin incrustation over the heated surfaces and the liquid is entirely evaporated by the time the lowest plate is reached. When a sufficient thickness of solid has accumulated on the plates in the tower, the liquid supply is stopped and heating is continued from outside the tower, a current of hot dry air being passed through so as to dry the deposited solid. A. R. Powell.

Filtering apparatus. J. B. Vernay (E.P. 266,719, 22.2.27. Conv., 24.2.26).—In a filter element for use in a thickening filter, the filtrate is withdrawn by suction from the lower part of the interior of the element, and the compressed air is admitted at the upper part.
B. M. Venables.

Apparatus for filtration. N. C. Christensen (U.S.P. 1,641,736, 6.9.27. Appl., 19.1.21).—A chamber closed at the sides and top is provided with suspended vertical partitions of filtering medium, arranged to form spaces which are entirely enclosed except for openings for the application of vacuum and pressure, and means are provided for dipping the lower end of the chamber into the mixture to be filtered. H. Holmes.

Filter. E. J. Sweetland (E.P. 275,333, 5.5.26).—A vacuum filter for removing a small quantity of solids from a large quantity of liquid is arranged so that it is operated in cycles of three stages, viz., collecting the cake, removing the cake by reversal of flow of filtrate, settling; the cake is thrown off into the same surrounding vessel from which the prefilt is taken, and is removed from it by a pump or conveyor at the bottom. The action of the filter is one of thickening only, the thick pulp being finally treated, if desired, in another filter. The filtering period may be 10 times as long as each of the other two, and the vacuum is preferably produced by a wet reversible pump, the discharge pipe being taken up vertically and holding just sufficient filtrate to form the necessary back-wash. The filter cloths are loose on their supporting members, so that they will distort largely on reversal. B. M. Venables.

Filters. E. J. Sweetland (E.P. 275,779, 23.7.26).—A number of disc-shaped filter leaves are attached to a hollow shaft and rotated within a casing, which when in use is pressure-tight, but can be slid on rails to permit inspection of the filter. Connexions for prefilt, wash water, air, and drainage are made to the casing, preferably through separate valves, but with a common detachable joint. The discs are divided into sectors, which are detachable radially, and each row of sectors is connected to a separate passage within the shaft. During the filtering stage the discs are rotated slowly. Discharge is effected by rotating them quickly in wash water.
B. M. Venables.

Spray drying. Industrial Associates Inc. (E.P. 272,859, 4.3.27. Conv., 18.6.26).—A spraying chamber is jacketed round the upper part by the incoming hot air and round the lower part by the outgoing moist air. A cloud of the liquid enters the inner sprayer chamber at the centre of the top, and is surrounded by a stream of the heated air. They move together downwards, the chilled moist air leaving by the lower circumference. There is also a circulation up the sides of less chilled air. The dried powder is deposited on the bottom, and is removed, *e.g.*, by rotating rakes. B. M. Venables.

Apparatus for atomising and drying liquids. A. Nyrop (E.P. 275,404, 21.7.26).—Air is admitted tangentially and in a downward direction to the upper circumference of the drying chamber, and the liquid is centrifugally sprayed through the centre of the top. The moist air is withdrawn at the centre of the bottom through a pipe which upstands somewhat. The dried powder is deposited on the bottom. The air may be admitted in puffs. B. M. Venables.

Atomiser for [corrosive] liquids. F. E. Martin (E.P. 271,035, 31.12.26. Conv., 12.5.26).—A mechanically driven centrifugal spraying device stated to be suitable for use in sulphuric acid chambers etc. is described. The liquid is flung off from, and the drops caught on the next of, several edges in succession, so that eventually all is reduced to fine mist.
B. M. Venables.

Apparatus for the production of freezing liquid. N. Dahl (E.P. 275,849, 18.12.26).—Brine is caused to flow down through a column of ice without any splashing or entrainment of air by supplying the brine above a grating surrounding a hopper which is kept full of ice and forms an upward extension of the ice column sufficient to keep ice sunk below the brine in the wider part of the column. Several columns are connected in series, the point of entry to each being slightly lower than the one before; by this means the upper level of the brine is automatically kept above the grating, and the final outlet temperature of the brine is very uniform.
B. M. Venables.

Pasteurisation of liquids containing gases. A./S. de Forenede Bryggerier (Austr. P. 105,348, 27.6.25).—Liquids charged with gases are pasteurised by heating them to the desired temperature in vessels with expansible walls; after cooling, the walls of the vessels are forced back to their original position by external pressure. Suitable vessels may be made of aluminium, the bottom being of soft aluminium and the remainder of the vessel of hard aluminium. A. R. Powell.

Method and device for conditioning gases as regards their temperature and humidity. Carrier Engineering Co., Ltd., and S. L. Groom (E.P. 276,214, 24.12.26).—The gas is preheated, then humidified by sprays, and the excess liquid from the sprays is recirculated. The conditioned gas passes over a thermostat which regulates the amount of preheating.
B. M. Venables.

Apparatus for heating air and/or other gases. F. A. Fahrenwald and H. E. Smith (E.P. 275,706, 6.5.26).—A continuously operating stove, *e.g.*, for heating the air for blast furnaces, is constructed with the air passing through metallic pipes which in the hotter zones may be of chromium alloys. Provision is made for expansion and contraction and for the use of the external casing of existing stoves. B. M. Venables.

Deodorising, cooling, and dehydrating fluids and apparatus therefor. H. L. Murray, Assr. to Te Aroha Dairy Co., Ltd. (U.S.P. 1,641,349, 6.9.27. Appl., 7.3.24. Conv., 14.8.23).—See E.P. 220,627; B., 1926, 74.

[Heat-insulated iron walls for] annular ovens or furnaces with rotary hearths. Trocknungs-, Verschwelungs- u. Vergasungs-G.m.b.H., L. Honigmann, and F. Bartling (E.P. 275,041, 2.10.26).

[Blocks for] furnaces having suspended arches. L. Morton and J. Hargrove (E.P. 275,008, 7.7.26).

[Automatic, reversible] absorption refrigerating apparatus. J. O. Boving (E.P. 276,088, 21.5.26).

Absorption refrigerating or heating machine. SIEMENS-SCHUCKERTWERKE G.M.B.H. (E.P. 251,251, 31.3.26. Conv., 23.4.25).

Absorption refrigerating machines. SULZER FRÈRES SOC. ANON. (E.P. 272,865, 3.5.27. Conv., 15.6.26).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Combustion tray for determination of heating value of coal. G. B. WATKINS and J. V. HUNN (Ind. Eng. Chem., 1927, **19**, 1020).—Incomplete combustion, due to loss of sample from the combustion tray, in determinations of the calorific value of coal is avoided by the use of an illium combustion tray $\frac{1}{2}$ in. deep, diam. at base $\frac{7}{8}$ in. and at top 1 in., with holes drilled round the side and half-way up. The coal sample is heaped into a cone in the tray and combustion conducted in the normal way. Oxygen is drawn in through the holes as combustion proceeds. R. BRIGHTMAN.

Propagation of a zone of combustion in powdered coal. III. Composition of the oxidation product. S. H. JENKINS and F. S. SINNATT (Fuel, 1927, **6**, 421—424; cf. B., 1924, 896).—The loss in weight due to the passage of a zone of combustion through a conical heap of powdered coal varied in different experiments from 5·2 to 12·7% for one coal and from 9·1 to 21·2% for another. In spite of this variation the composition of the oxidation product from the same coal was almost constant. The most marked changes in composition on combustion were the increase in the oxygen content (*e.g.*, from 7·29 to 16·72%, or from 9·51 to 18·31%) and the complete loss of caking power.

A. B. MANNING.

Total carbon in coal determined by analysis of gas from bomb calorimeter. G. B. WATKINS (Ind. Eng. Chem., 1927, **19**, 1052—1054).—A sample of coal is exploded in a Mahler bomb under an oxygen pressure of 20 atm., and the calorific value determined. The gaseous products of combustion are then expanded into a mercury-sealed gasometer and the carbon dioxide content is determined. The gasometer of 9 litres capacity was constructed of two concentric steel tubes so as to use a minimum quantity of mercury. It was calibrated to read at 100 c.c. intervals. Tests on standard coal samples, benzoic acid, and sucrose show values for total carbon accurate to 0·1%. C. IRWIN.

Nitrogen as a catalyst in the determination of sulphur in coal by the bomb-washing method. J. F. KOHOUT (Ind. Eng. Chem., 1927, **19**, 1065—1066).—It is shown that increasing the nitrogen content in the gases in a bomb calorimeter from the usual 5% to 10—15% generally caused a slight increase in the apparent sulphur content of the coal tested, as determined by washing out the bomb and weighing as barium sulphate. This is attributed to the increased catalytic action of oxides of nitrogen in completing oxidation to sulphuric acid, and brings the results more in harmony with the Eschka or sodium peroxide methods.

C. IRWIN.

Resins in coal and their effect upon its properties. C. COCKRAM and R. V. WHEELER (Fuel, 1927, **6**, 425—428).—The small yellow rods, varying in length up to 0·02 in., which appear in the residue from the regulated oxidation of certain coals, *e.g.*, Hamstead vitrain, after removal of the regenerated ulmins by dissolving in dilute sodium hydroxide, are shown by their solubility in chloroform and their composition to be resin inclusions. Photomicrographs show that the resin rods exist in the coal in the positions which they had occupied in the woody tissue from which the vitrain was formed. The resin is extracted from the coal by treatment with pyridine and chloroform, and therefore appears in the γ-compounds; the yields of γ-compounds from a "normal" and a "resinous" sample of Parkgate clarain were 4·9 and 7·2%, respectively. In an assay test at 600° the tar yield from the resinous clarain (16·6%) was much higher than that from the normal clarain (12·1%). Although the coke from the former was highly swollen, the degree of swelling was less than that in an average clarain from the same seam.

A. B. MANNING.

Inherent ash of coal. R. A. MOTT and R. V. WHEELER (Fuel, 1927, **6**, 416—420).—The amount and composition of the "inherent" and the "adventitious" ash of a number of samples of clarain and durain from the Parkgate seam, of durain from the Nottinghamshire Top Hard seam, and two samples of vitrain, have been determined. The coal substance containing the inherent ash was separated from the adventitious ash by suspension of the very finely-ground material in water and flocculation of the coal by the addition of paraffin oil. Comparison of the SiO_2/Al_2O_3 ratios shows a definite difference in composition between the inherent and adventitious mineral matter in the samples. The differences are somewhat greater for the durains than for the clarains, and are, moreover, in the opposite direction. Comparison with analyses of the ash of modern non-flowering plants indicates that these are poorer in silica than their earlier prototypes. The iron present in clarains is probably chiefly adventitious. The presence of nickel and zinc in the clarain and durain of one seam section has been established. No close relationship could be established between the composition of the adventitious ash in the coal and that of the shale occurring as a dirt band in the seam or composing the floor of the bed. A. B. MANNING.

Determination of the porosity of coke. N. A. ROSS (Gas World, 1927, **87**, Coking Sect., 97—98).—In determining the volume of a piece of coke by immersion in water considerable errors (up to 6%) may be caused by the coke taking up water. More consistent and accurate results are obtained by using mercury (cf. Greenwood and Cobb, J.S.C.I., 1922, **41**, 181 T). The real sp. gr. must be determined on material so finely ground that no closed pores remain in it; on the other hand the degree of subdivision used when determining the sp. gr. of the material plus closed pores must not be such as to affect appreciably the coke structure.

A. B. MANNING.

Lustre carbon as initial member of the black, crystalline carbon series. K. A. HOFMANN and U. HOFMANN (Ber., 1926, **59**, 2433—2444; cf. Hofmann and Röchling, B., 1923, 1130 A).—Highly lustrous carbon is

most certainly prepared by passing the vapours of light petroleum, initial b.p. 90°, mixed with nitrogen, oxygen, water vapour, carbon monoxide, or carbon dioxide into a crucible heated electrically at 750—800°. Decomposition occurs thermally at the porcelain surface, and formation of soot in the gas is avoided. A uniform, highly lustrous, completely non-transparent, black layer is formed on smooth surfaces of porcelain, quartz, fluorspar, silicon carbide, diamond, zinc blende, gold, or silver, whereas a grey layer is produced on rough surfaces of unglazed porcelain, calcined magnesia or asbestos, electrode retort carbon, or Acheson graphite; the grey material is also formed on smooth surfaces above 860°. Prolonged exposure of diamond to hydrocarbon vapours at 700° and 500 atm. does not lead to growth of the crystal. Lustre carbon has d^{16} 1·86±0·02, the density increasing with increasing temperature of formation. After prolonged heating at 850° in an atmosphere of dry hydrogen it appears to be pure carbon. It is not capable of absorbing phenol, thus resembling graphitic carbon. Towards oxygen, water vapour, carbon dioxide, sodium sulphate, and potassium cyanate lustre carbon is much more stable than sugar carbon prepared at the same temperature, and resembles closely retort graphite. Both varieties are more active than is natural graphite. Röntgen spectrographic observations show that graphite, retort graphite, lustre carbon, and sugar charcoal are all crystalline in structure, and that the size of the crystallites diminishes in the given order.

H. Wren.

Composition of benzol from carburetted water-gas. W. A. Voss (J.S.C.I., 1927, **46**, 373—375 T).—Benzol extracted from carburetted water-gas by means of active charcoal is compared with the benzol similarly extracted from coal-gas produced in horizontal retorts. The benzol from carburetted water-gas contained only 0·73% of paraffins, but contained 12% of unsaturated hydrocarbons, which rendered the purification of the spirit difficult. The benzol from coal-gas contained 2·57% of paraffins, but only 5% of unsaturated hydrocarbons. Rectified benzol from carburetted water-gas is richer in toluene than the benzol from coal-gas. The sample examined contained 32·4% of toluene compared with 17·97% in the water-gas sample. The difficulty in removing the unsaturated compounds from carburetted water-gas benzol is shown graphically, the loss in volume being plotted against the degree of acid washing. With adequate washing a satisfactory benzol can be obtained from carburetted water-gas.

Determination of carbon monoxide, hydrogen, and methane in air containing ethylene, applicable to the analysis of the products of explosion of blasting explosives. J. Thorburn (J.S.C.I., 1927, **46**, 355—358 T).—One litre of the gas to be analysed is automatically displaced by mercury from a pipette and passed through the purification and reaction systems. Carbon dioxide is removed by caustic potash and ethylene by 20—25% oleum. Sulphur trioxide from the oleum is absorbed in caustic potash, and moisture is removed by calcium chloride followed by phosphorus pentoxide. Carbon monoxide is then oxidised by iodine pentoxide in a U-tube heated at 150° in an oil bath, and the percentage determined from the quantities of iodine and carbon dioxide formed, the iodine being absorbed in a 15% potassium iodide solution and the carbon dioxide in $N/9$-barium hydroxide solution. The gas is dried again and the hydrogen is oxidised, by the oxygen present in the sample, over palladium asbestos heated in an electric tube furnace with thermostatic control at 255 ± 5°; the water formed is caught in a weighed tube of special design containing phosphorus pentoxide, and from this the percentage of hydrogen is calculated. The gas is next passed through $N/9$-barium hydroxide solution to determine any accidental oxidation of methane at this stage, and after being dried again it is passed over copper oxide heated in a quartz tube to a red heat in a small gas furnace to burn the methane, the percentage of which is calculated both from the amount of water and that of carbon dioxide formed, these substances being absorbed as already indicated. As detailed, the method is applicable to mixtures containing 0—4% of each of the components to be determined, and 0—1% of ethylene. It is used when large samples of gas are available if special accuracy is required.

Ignition of gases by hot wires. W. C. F. Shepherd and R. V. Wheeler (Safety in Mines Res. Bd., Paper No. 36, 1927, 26 pp.).—A wide range of mixtures of methane and air can be ignited by the glowing (tungsten) filament of a miners' electric lamp bulb, when the latter is broken in such a way as to leave the filament intact. Determinations have been made of the minimum current which, when caused suddenly to flow through a platinum or tungsten wire, eventually causes ignition of methane–air mixtures, and of the time interval between completing the circuit and ignition. For a platinum wire of given diameter the minimum igniting current is constant over the range of ignitable mixtures, varying from 1·65 amp. for a wire of 0·1 mm. diam. (ignitable range 4·55 to 7·50% of methane) to 6·6 amp. for a wire 0·3 mm. diam. (ignitable range 4·30—15·2% of methane). Platinum wires less than about 0·1 mm. in diameter fuse before causing ignition of any mixture. Some surface combustion occurs with platinum wires in all methane–air mixtures. Other conditions remaining constant, the time interval before ignition passes through a maximum value as the composition of the mixture is varied, the corresponding composition coinciding with that of the mixture of maximum calorific value (9·45% of methane). The higher temperature of the wire due to the greater heat developed by surface combustion in such a mixture causes an increased convection effect with a resulting minimum time of contact of the gas with the wire. When a tungsten wire is heated in an atmosphere containing oxygen it is oxidised, and its temperature rises until it burns with a small bright flame, which always ignites an inflammable methane–air mixture; the most readily ignitable mixture contains about 8% of methane. It is therefore recommended that an automatic device, which breaks the electric circuit as soon as the outer protecting glass is broken, should be incorporated in the design of a miner's electric hand-lamp.

A. B. Manning.

Rôle of lead sulphide in the sweetening of petroleum distillates and chemistry of the mercaptans. J. C. Morrell and W. F. Faragher (Ind. Eng. Chem., 1927, **19**, 1045—1049).—Solutions of pure

ethyl, *n*-butyl, *iso*amyl, heptyl, and benzyl mercaptans in refined, sulphur-free Pennsylvania naphtha were treated with solutions of sodium hydroxide in various mixtures with lead sulphide and sulphur in a stream of nitrogen or oxygen. Panhandle gasoline (Amarillo, Texas) containing 0·05% of mercaptans and washed free from hydrogen sulphide was similarly treated. With oxygen alone no sweetening occurred, and the mercaptans are not oxidised under the conditions of the test. Agitation with sodium hydroxide and sulphur in a current of nitrogen sweetened the mercaptan solutions, but not the Panhandle gasoline. Sodium mercaptides are formed first and then the disulphide (cf. Holmberg, A., 1908, i, 308): in the case of *iso*amyl mercaptan the use of a current of oxygen in place of nitrogen considerably reduced the time required for sweetening. Lead sulphide suspended in water or in the oil did not effect any sweetening, and hence lead sulphide does not catalyse the oxidation of mercaptans (cf. Wendt and Diggs, B., 1924, 1002). Mixtures of lead sulphide and sodium hydroxide agitated in a current of oxygen effected sweetening in all cases except those of *iso*amyl mercaptan and the gasoline, lead mercaptides being formed in every case. Sweetening also occurred in a current of nitrogen containing 4% of oxygen, sulphate ions being formed in all cases. Lead sulphide plays a minor part in the stability of lead mercaptide dispersions, functioning similarly to fuller's earth and charcoal, but being less active. Agitation with a solution of sodium plumbite in either nitrogen or oxygen effected sweetening exactly as in the case of a mixture of lead sulphide and sodium hydroxide, slightly better results being obtained with a current of oxygen. There action, $2(RS)_2Pb + O_2 \rightarrow 2RS{\cdot}SR + 2PbO$, takes place whether the sodium plumbite or the lead sulphide and sodium hydroxide are used. Panhandle gasoline cannot be sweetened unless lead sulphide and oxygen as well as sodium plumbite are present. The nature of the lead sulphide is also a factor. Thus, no sweetening action was observed with galena, and lead sulphide precipitated from lead acetate solution with hydrogen sulphide was less effective than that obtained by passing the gas over lead dioxide, the sweetening activity depending mainly on the rate of oxidation of the sulphide, and the rate of sweetening on the oxygen concentration, being greatest in ozonised air. No sweetening action occurs in pure nitrogen. In the oxidation of lead sulphide in sodium hydroxide suspension with oxygen, one mol. of sodium sulphate is formed per mol. of sodium plumbite. Mercury, silver, copper, and nickel sulphides exert a similar sweetening action on benzyl mercaptan, and nickel sulphide in the case of butyl mercaptan also, sulphate being formed in all cases. Addition of sulphur to the mixture of butyl mercaptan and suspended sulphides in sodium hydroxide produced sweetening in every case, sulphate ion being again formed together with the mercaptide of the metal and subsequently the disulphide. R. Brightman.

Decomposition of waxes. Mailhe.—See XII.

Patents.

Coke oven. C. Still (E.P. 275,914, 12.5.27).—A combination of the arrangement for the uniform heating of coke ovens described in E.P. 221,697 (B., 1924, 900 with means for withdrawing the gaseous distillation products at or near the bottom of the coking chamber avoids undue cracking of the gaseous products, and at the same time gives a good quality coke. If the chamber is unevenly heated and the gaseous products are withdrawn from the bottom, cracking still occurs due to the rapid overheating of the lower part of the charge. In the process now described it is preferable so to distribute the heating that the temperature of the upper part of the wall is slightly higher than that of the lower part. A. B. Manning.

Carbonisation at low temperature of hydrocarbonaceous material. C. Ab-Der-Halden (E.P. 263,197, 20.12.26. Conv., 21.12.25).—A retort for the low-temperature carbonisation of fuels consists of two (or if necessary more) pans fixed to a vertical axis which can be rotated. The material is carbonised in the lower pan, which rotates in an enclosure heated externally by the gases from a fire grate, and is so designed that the combustion gases cannot penetrate into the enclosure. The hot flue gases then pass through the enclosure in which the upper pan rotates, and in which the material undergoes a preliminary drying. By means of inclined scrapers the material is made to pass along a spiral path from the periphery to the centre of the upper pan, whence it descends through a passage to the lower pan, which it traverses similarly in the reverse direction until discharged through a passage at the periphery. The inclination of the scrapers controlling the circulation of the material can be adjusted from outside the retort. A. B. Manning.

Carbonising, distilling, and gasifying solid fuel. G. W. Wallace (U.S.P. 1,639,390—1 and 1,639,417, 16.8.27. Appl., [A] 18.5.25, [B] 13.1.26, [C] 24.5.26).—(A) The fuel passes along a horizontal conduit and is met by a stream of hot gases issuing from a jet, whereby sudden local heating is effected. The heating means are adapted to the maintenance of a substantially stationary carbonising zone in the reaction chamber. (B) The fuel is treated in substantially the same manner as in (A), but a flame of burning gases issues from the jet in place of hot gases, and the material heated thereby is gasified by means of a draught current. (C) Coke and by-products are produced by a process similar to that described in (B). C. O. Harvey.

Distillation and coking of carbonaceous matter. L. Honigmann and F. Bartling (U.S.P. 1,640,502, 30.8.27. Appl., 7.1.27. Conv. 26.11.25).—Solid carbonaceous material is distilled and coked on annular revolving hearths in a layer less than 1 cm. thick. C. O. Harvey.

Production of ammonium nitrate and nitrite etc. and generation of additional power from the combustion gases in internal-combustion engines. E. R. Besemfelder (G.P. 440,622, 16.4.25).—The combustion chamber is provided with contact material to promote formation of oxides of nitrogen, and the exhaust gases are cooled by the evaporation of a liquid in a closed exhaust system provided with a release valve in connexion with a turbine operated by the escaping gases. The nitrous and nitric acids formed in the process are neutralised with ammonia. L. A. Coles.

Manufacture of active carbon. I. G. Farbenind. A.-G., Assees. of Farbenfabr. vorm. F. Bayer & Co. (E.P. 251,636, 28.4.26. Conv., 29.4.25).—Cellulose carbonaceous material (wood, peat, lignite, etc.) is treated with sulphuric acid and heated in a furnace at about 750°, the carbon produced being further heated at about 900° while steam is passed through the furnace.
W. G. Carey.

Manufacture of activated carbon. G. W. Wallace (U.S.P. 1,639,356, 16.8.27. Appl., 13.1.26).—A moving pervious column of material is carbonised by hot flame gases impinging on and moving in counter-current to the material, whereby a stationary zone of carbonisation is maintained. The hot carbonised material is partially oxidised by another draught current. C. O. Harvey.

Carbonaceous material and process for making same. Roessler & Hasslacher Chem. Co. (E.P. 251,982. 30.4.26. Conv., 9.5.25).—A dense carbon of low porosity and suitable for electrodes etc. is made by mixing finely-divided graphite with a binder and carbonising under pressure. Thus a mixture of 80% of graphite and 20% of hard pitch was pressed at 250° in a steel mould which was then heated at the temperature of decomposition of the pitch. The carbon was removed from the mould and heated at 1000°; the resulting product has an apparent sp. gr. of 1·8—1·9.
A. C. Monkhouse.

Production of highly-active blood charcoal. Chem. Fabr. auf Actien (vorm. E. Schering) (G.P. 440,769, 14.8.25).—Blood or blood serum mixed with ammonium sulphate is carbonised in the presence of an excess of alkali carbonate. L. A. Coles.

Process and apparatus for the manufacture of gas from heavy oils. C. Chilovsky (E.P. 255,423, 19.5.26. Conv., 16.7.25).—A process for the continuous complete conversion of oils into combustible gases, in which no fouling of the catalyst occurs, consists in passing the oil (with or without admixture with steam, metallic chlorides in the case of animal and vegetable oils, coal in colloidal suspension, etc.) through a vaporiser, mixing the vapours with heated air (or oxygen) in proportions of about 2 and not more than 3 g. of oil to 5 litres of air, and allowing partial combustion to take place in a heat-insulated cylindrical chamber whereby the mixture attains a temperature of 700—1000° prior to passing through a catalyst (steel rivets, molten cast iron, etc.). The hot gases produced may be used for preheating the oil and air, and the process may be carried out under partial vacuum. C. O. Harvey.

Manufacture of gas from heavy oils. C. Chilovsky (E.P. 271,899, 19.5.26. Conv., 16.7.26. Addn. to E.P. 183,828; B., 1924, 46).—The process of the main patent is improved by passing the atomised and gasified heavy oil over a metallic or non-metallic catalyst at 700—900°. The whole of the heat required for the process may be generated in the combustion chamber, or the latter may be maintained at a lower temperature (400—600°) and the heat then required for maintaining the temperature of the catalyst at 700—900° may be produced by the subsequent introduction of additional air and combustion of a further portion of the oil. Steam may be added to the mixture to prevent clogging of the catalyst. Metallic chlorides are added to vegetable and animal oils, and also, if desired, to mineral oils, before subjecting them to the process. A. B. Manning.

Desulphurisation of fuel gases. E. Will (Austr.P. 105,362, 28.6.23. Conv., 15.5.23).—The gases are passed through strongly heated chambers, fitted completely or partly with chequer work of basic material, between which and the acidic material of the chamber walls there is a layer of magnesite or magnesite brick.
A. B. Manning.

Gas scrubber. A. C. D. Duchemin (E.P. 263,794, 20.12.26. Conv., 31.12.25).—A horizontal gas scrubber is divided by partitions, slightly inclined in the direction of the gas flow, into a series of chambers containing filling material, and has empty expanding spaces between successive chambers for equalising the gas flow. The scrubbing liquid is pumped from the bottom of each chamber to a spraying device at the top, and at the same time, by the aid of weirs of different heights, is circulated through the lower part of the apparatus in a direction opposite to that of the gas flow.
A. B. Manning.

Regeneration of contact masses for the catalytic hydrogenation of carbon oxides. G. Patart (E.P. 252,361, 12.5.26. Conv., 25.5.25. Cf. F.P. 599,588; B., 1926, 351).—The contact masses containing zinc chromate or tungstate etc. used as catalysts in the synthesis of methyl alcohol, and which have lost activity owing to the accumulation thereon of sulphur compounds, can be regenerated by calcination in an oxidising atmosphere, as, for example, by heating to redness in an open crucible. A. B. Manning.

Increasing the safety of porous and absorbent materials used for storing explosive combustion gases [*e.g.*, acetylene]. R. Heppner (E.P. 264,846, 20.1.27. Conv., 20.1.26. Cf. G.P. 420,150; B., 1926, 230).—Absorbent material used for storing acetylene etc. is treated with inorganic or organic salts which volatilise at high temperatures, consuming heat and so preventing further decomposition of the gas, or which decompose into gases exerting a fire-choking action. Pumice stone meal, metal oxides, etc. mixed with adhesives such as oils, fats, or resins insoluble in the gas solvent, and incapable of reacting with the gas, are added to the absorbent material to improve its homogeneity.
W. G. Carey.

Production of liquid hydrocarbons and derivatives thereof from coal, tar, etc. I. G. Farbenind. A.-G. (E.P. 249,155, 13.3.26. Conv., 14.3.25. Addn. to E.P. 247,217; B., 1927, 601).—In the process of converting coal, tar, etc. into liquid hydrocarbons by treatment with reducing gases under pressure and at high temperatures, with or without a catalyst, the hot surfaces of the apparatus which come into contact with the high-pressure gases are made of a metal which does not react with carbon monoxide. Suitable metals are copper, silver, aluminium, chromium, manganese, etc., and special steels containing 10—20% of manganese, chromium, tungsten, etc. A. B. Manning.

Process and apparatus for the cracking of oils. S. Seelig (E.P. 268,323, 11.3.27. Conv., 25.3.26).—In a process for cracking oils by passage through molten

metal, the interior of the vertical cylindrical chamber in which the operation is conducted is protected from fluctuations of temperature by the introduction of a helical tube which may be evacuated or may contain air or steam, and which serves as a heat-insulating medium. The gases passing through the tube may be led into the vapour space above the molten metal, thereby assisting in the removal of the cracking products. C. O. Harvey.

Process and apparatus for splitting fluid hydrocarbons. C. Longhi (E.P. 275,281, 1.3.26).—The method of cracking oils by means of an electric arc immersed in the oil (cf. E.P. 248,830; B., 1926, 525) is modified by providing a rotating central electrode, carrying a number of arcing points and a corresponding number of arcing points attached to the body of the apparatus, whereby the arcs are rapidly made, elongated, quenched, and remade. When the speed of rotation is great enough, the arcs become in effect a rotating disc, and in such circumstances it is advisable to have both electrodes rotatable. C. O. Harvey.

Treatment of cracking or sludge residues. N. V. de Bataafsche Petroleum Maatschappij, and F. R. Moser (E.P. 270,274, 13.4.27. Conv., 27.4.26).—The residues are treated with small quantities of substances having an acid reaction (*e.g.*, ferrous chloride, aluminium chloride or sulphate, sulphur chloride, organic acid chlorides, etc.) with or without the addition of a little water, and are then heated at temperatures of 100—200°, whereby asphaltic and coke-like substances are rendered easily removable by filtration, centrifuging, etc. Viscous residues are diluted with crude oil, fuel oil, or gas oil before treatment. C. O. Harvey.

Process and apparatus for cracking oil. G. Egloff, Assr. to Universal Oil Products Co. (U.S.P. 1,638,115, 9.8.27. Appl., 13.12.20. Renewed 24.3.27). —The oil to be cracked is passed in a thin stream under pressure through a heating chamber and thence in a substantially liquid state to an expansion chamber, the upper part of which is heated internally, and which carries a reflux condenser whereby less volatile products are returned for retreatment, the vapours being condensed and collected under pressure. C. O. Harvey.

Method and apparatus for cracking oil. F. M. Hess (U.S.P. 1,638,335, 9.8.27. Appl., 24.1.23).—The oil flows in a thin stream over inclined heating plates situated in a pressure still, the residual uncracked oil passing to a producer wherein it is converted into a gas rich in hydrogen which is discharged into the still and mixed with the vapours of distillation.
C. O. Harvey.

Process for cracking oil. G. Egloff, Assr. to Universal Oil Products Co. (U.S.P. 1,638,093, 9.8.27. Appl., 20.12.20. Renewed 3.1.27).—The oil passes through a cracking coil to an expansion chamber, the latter being connected with a larger expansion chamber for receiving evolved vapours prior to dephlegmation and condensation. The operation is carried out under pressure. C. O. Harvey.

Apparatus for distilling oil. J. W. Coast, jun., Assr. to Doherty Research Co. (U.S.P. 1,639,327, 16.8.27. Appl., 3.11.20).—The interior heated surface of a horizontal cylindrical still is kept clean by means of a scraping member carried on an endless chain.
C. O. Harvey.

Apparatus for the distillation of oil. F. T. Manley, Assr. to Texas Co. (U.S.P. 1,640,202, 23.8.27. Appl., 26.10.18. Cf. U.S.P. 1,428,338—9; B., 1922, 850 a).—A horizontal still is provided with a vapour space situated above the level of the oil, external heat being applied around practically the entire periphery of both liquid and vapour spaces, and the latter being lagged in order to avoid overheating of the vapours, while maintaining a uniform cracking temperature. C. O. Harvey.

Art of distilling oils. F. A. Howard and N. E. Loomis, Assrs. to Standard Development Co. (U.S.P. 1,640,938, 30.8.27. Appl., 10.4.22).—The oil passes continuously through the following steps in succession: volatile constituents are removed by heating at ordinary pressure; the residual oil is cracked under pressure and the vapours are removed after reduction of pressure; the residue is mixed with fresh incoming oil.
C. O. Harvey.

Continuous treatment of hydrocarbons [with liquid sulphur dioxide]. O. Y. Imray. From Allgem. Ges. für Chem. Ind. m.b.H. (E.P. 275,884, 2.3.27).—The process is a modification of that of E.P. 114,845 (B., 1920, 98 a). The oil ascends through a mixer, passing counter-current to the liquid sulphur dioxide, and the resulting extract and raffinate are subjected to a continuous evaporation process divided into a number of pressure stages. In the first stage the sulphur dioxide is removed principally by the action of heat, in the next successive stages by the combined action of heat and reduced pressure, and in the last stage preferably only by the application of a comparatively high vacuum, so that the gases evolved in the first stage flow into the condenser tank by their own pressure, whilst the gases evolved in the subsequent stages are brought up to the pressure of the tank by compressors. The gases passing from the evaporators are freed before compression from any light hydrocarbons by suitable cooling. A. B. Manning.

Refining hydrocarbon oils. P. McMichael, Assr. to Hydrocarbon Refinishing Process Co., Inc. (U.S.P. 1,641,546, 6.9.27. Appl., 25.5.25).—Cracked hydrocarbon oils are treated with sulphur dioxide and then with an absorbent material. C. O. Harvey.

Purification of petroleum products. S. J. Dickey and R. C. Wheeler, Assrs. to Gen. Petroleum Corp. of California (U.S.P. 1,639,988, 23.8.27. Appl., 19.4.24). —The sludge is removed from an acid-treated distillate by filtration through a pack of dried sand, the sand being subsequently cleaned *in situ* by means of steam and dried by a current of air. C. O. Harvey.

Apparatus for purifying used mineral lubricating oil. R. E. Langston, Assr. to Wayne Co. (U.S.P. 1,640,415, 30.8.27. Appl., 27.9.21).—The oil is purified and freed from volatile matter by being heated to a temperature substantially above 100° in a jacketed vessel heated by means of steam coils, which extend from the jacket into the vessel and open thereinto.
C. O. Harvey.

Fuel and method of operating internal-combustion engines with same. H. F. Parker (E.P. 275,334, 5.5.26).—Aromatic hydrocarbons extracted from the kerosene fraction of petroleum distillates by means of liquid sulphur dioxide are added to untreated kerosene until their content is greater than 50%. The flash point of the resulting fuel is above 38°, and the detonating property of the original kerosene is reduced. In airships having an internal-combustion engine with a compression ratio of not less than 5 : 1, this fuel can be used in conjunction with hydrogen to obtain a high water-vapour content in the exhaust gases. A. C. Monkhouse.

Lubricant for automotive engines. L. B. Lockhart, Assr. to Standard Oil Co. (U.S.P. 1,639,347, 16.8.27. Appl., 20.2.26).—An oil for use in engines of the type in which liquid fuel enters the oil from the cylinders contains 7—15% of fractions of b.p. 149—238°, and has a flash point above 66°, a Saybolt viscosity of at least 140 at 37·7°, and a relatively low loss in viscosity during use. C. O. Harvey.

Production of active carbon. P. Lueg, J. Drucker, and H. Thienemann, Assrs. to I. G. Farbenind. A.-G. (U.S.P. 1,641,281, 6.9.27. Appl., 7.11.25. Conv., 29.4.25).—See E.P. 251,636 ; preceding.

Conversion of heavy petroleum oils into lighter oils. V. W. Northrup, Assr. to Petroleum Hydrogenation Co. of America, Inc. (U.S.P. 1,642,624, 13.9.27. Appl., 20.11.24. Renewed 8.2.27).—See E.P. 275,120 ; B., 1927, 741.

Flotation separation (E.P. 275,778).—See I.

By-products from coke ovens (E.P. 266,004).—See III.

III.—TAR AND TAR PRODUCTS.

Raschig continuous tar distillation plant of the Nippon Seikosho. M. Fukunaga (J. Fuel Soc. Japan, 1927, 6, 58—60).—The tar treated (30—35 tons/day) has *d* 1·20, 2·4% of moisture, and 9·23% of free carbon, the products being 2·61% of water and 0·73% of light oil (from No. 1 still) ; 1·18% of carbolic oil and 2·88% of naphthalene oil (from No. 2 still) ; 20·45% of creosote oil and 4·0% of anthracene oil (from No. 3 still) ; and 66·0% of pitch. The light oil and carbolic oil contain, respectively, 10·1% and 25·7% of phenols, and the pitch has *d* 1·23, m.p. 64°, free carbon 16·47%, ash 0·13%, and solubility in carbon disulphide 82·64%. W. T. K. Braunholtz.

Dehydrogenation [of tar products etc.] by means of sulphur. W. Friedmann (Brennstoff-Chem., 1927, 8, 257—259).—Sulphur reacts violently with indene at its b.p. yielding a series of sulphur compounds, the two substances $C_{18}H_{12}S$ (di-indenylthiophen) and $C_{36}H_{24}S$ being obtained in a crystalline form. Di-indenylthiophen is also obtained by the action of sulphur on hydrindene, which itself is simultaneously readily converted into indene. Tetrahydronaphthalene is almost quantitatively dehydrogenated when heated with sulphur under pressure at 240—250°, dinaphthylthiophen (m.p. 247—248°) being also formed. The determination of hydrogenated compounds in tars by means of sulphur, whilst of value in the case of benzene and naphthalene derivatives, is less reliable in the case of those of unsaturated and highly reactive compounds, owing to their tendency to form products containing nuclear sulphur (*e.g.*, thiophen derivatives). W. T. K. Braunholtz.

Deodorisation and decoloration of the phenols in lignite oils. A. Mailhe (Bull. Soc. chim., 1927, [iv], 41, 1062—1064).—The mixed phenols (b.p. 190—230°) extracted from the black heavy oils which are obtained on rapid distillation of the tar produced when certain lignites are dry-distilled contain thiophenic compounds with a disagreeable odour, and, because of impurities, they darken on keeping. If they are warmed with stannous chloride, washed, and distilled, a product is obtained from which almost pure cresols and *m*-xylenol may be isolated, containing only 0·5—0·6% S. Apparently the thiophenic compounds decompose into hydrogen sulphide and hydrocarbons, and the coloured compounds polymerise to a thick liquid which settles with the stannous chloride at the bottom of the vessel. Other chlorides are less efficient. S. K. Tweedy.

Patents.

Recovery of by-products [phenols] in coke oven or like plants. C. Still, Assee. of A. Weindel (E.P. 266,004, 14.2.27. Conv., 15.2.26).—The major portion of the phenols present in the ammonia liquor is found in the liquor from the condensers. This condensate only is treated for the recovery of phenols, or in cases where the gas passes through a series of coolers the fractions of the condensate containing the greater portion of the phenols. The condensate is freed from tar and extracted at ordinary or raised temperatures with an organic solvent, *e.g.*, solvent naphtha or high-boiling tar oil. The phenols are recovered from the solvent by washing with alkali. Any solvent remaining in the washed condensate is removed and the condensate is mixed with the ammonia liquor from the washers for the subsequent recovery of ammonia. A. C. Monkhouse.

Producing mixed phenols and related compounds [from coal tar]. R. Cross (E.P. 275,313, 3.5.26).—Coal tar or its heavier distillates are " cracked " at a pressure above 350 lb./sq. in. at 370—540° by passing through coils situated in a furnace and then through a reaction chamber. After discharge, the pressure is reduced and the fractions are separated in an evaporator, the volatile vapours passing through a dephlegmator and condenser. The phenols are recovered from the distillate by extracting with caustic soda and acidifying the extract ; their distillation range is 65—232°. The yield of phenol is increased by mixing with the coal tar liquids containing hydroxyl groups, *e.g.*, glycols, glycerols, and alcohols. A. C. Monkhouse.

Purification of tar acid-bearing oils. Combustion Utilities Corp., Assees. of S. Caplan (E.P. 276,216, 30.12.26. Conv., 22.9.26).—The undesirable coloration developed in some tar products (disinfectants, animal dips, etc.) is stated to be due to the presence in the original tar oils of strongly reducing *o*-dihydric compounds similar to, but not including, pyrocatechol. These are removed from the oil without substantial loss of tar acids by agitating with hot aqueous solutions of borax, the latter being subsequently recovered by adding a soluble ferric salt and an alkaline agent such as lime,

whereby precipitation of the colour-forming substances is effected. C. O. HARVEY.

Vulcanisation of tar for road-making purposes. MITTELDEUTSCHE HARTSTEIN-IND. A.-G. (F.P. 610,572, 3.2.26. Conv., 7.8.25).—The tar is refluxed at 120—200° with up to 25%, usually 5—15%, of sulphur, care being taken to avoid loss of the more volatile hydrocarbons, which preserve the vulcanised tar from oxidation by the air and so prevent loss of fluidity. A. B. MANNING.

Liquid hydrocarbons from tar etc. (E.P. 249,155).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Electrolytic reduction of vat dyes. F. BRYANS and F. M. ROWE (J.S.C.I., 1927, **46**, 335—338 T).—With the exception of flavanthrene, the reduction by electrolytic hydrogen of anthraquinone, indigotin, and certain anthraquinone vat dyes is exceedingly inefficient. The best results are obtained by the use of a high temperature, a lead cathode, a 5% sodium hydroxide suspension, a current density of 0·2 amp. per dm.^{2}, and the dyestuff in a fine state of division. The effect of additions such as alcohol, phenol, arsenious oxide, antimony pentoxide, sodium bisulphite, and quinones is shown. A repetition of the work of Binz and Schädel (B., 1912, 329) on the reduction of indigotin by electrolytically separated metals is given.

Naphthalenesulphonic acids. VIII. Hydrolysis of naphthalene-1 : 5-disulphonic acid. D. F. J. LYNCH and J. T. SCANLAN (Ind. Eng. Chem., 1927, **19**, 1010—1012).—The hydrolysis of pure naphthalene-1 : 5-disulphonic acid when heated in sealed tubes at temperatures varying from 100° to 220° and concentrations of sulphuric acid ranging from 1% to 85% closely resembles that of naphthalene-1 : 6-disulphonic acid (B., 1927, 324). Hydrolysis to naphthalene takes place with low concentrations of sulphuric acid, being 99—100% at 220° with 1—9% sulphuric acid, at 200° with 22% acid, and 180° with 42% acid. With higher concentrations of acid, the 1 : 5-acid is converted into other isomerides, identical with those formed from the 1 : 6-disulphonic acid at the same temperature. With the 1 : 5-acid, however, conversion also takes place at lower temperatures and in less concentrated acid than are required with the 1 : 6-acid. Although conversion into the 1 : 6-acid takes place it does not always do so under the conditions in which the 1 : 6-acid is stable. Thus, at 140°, whilst with 73% sulphuric acid the 1 : 5-acid is converted into the 1 : 6-acid and a trace of the 2 : 7-acid, in 77% sulphuric acid the 1 : 5-acid is completely converted into the 2 : 7-acid, the 1 : 6-acid being stable under these conditions. The reaction is thus not intramolecular, but probably one of resulphonation of the naphthalene produced by hydrolysis. In all cases the isomeric disulphonic acids formed are those which would be produced by direct sulphonation of naphthalene under identical conditions of temperature and concentration. R. BRIGHTMAN.

PATENTS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G., Assees. of FARBENFABR. VORM. F. BAYER & Co. (E.P. 252,182, 12.5.26. Conv., 12.5.25. Addn. to E.P. 229,330; B., 1925, 875).—The dyes of the prior patent are made by coupling the diazo compounds with alkyl- or aralkyl-aminonaphtholsulphonic acids and treating the products with arylsulphonyl chlorides in presence of acid-binding agents (sodium carbonate). C. HOLLINS.

Manufacture of new azine dyes and intermediate products. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 275,724, 12.5.26).—Violet to greenish-blue azine dyes of the Wool Fast Blue type are obtained by using 1 : 3 - di-(*p* - alkoxyphenylamino)naphthalenesul - phonic acids in place of the usual 1 : 3-diphenyl- or ditolyl-amino-compounds. The products show enhanced light-fastness. 1 : 3-*Di*-p-*anisylaminonaphthalene*-8-*sulphonic acid*, prepared from sodium hydrogen α-naphthylamine-3 : 8-disulphonate and *p*-anisidine and its hydrochloride at 105—150°, is oxidised together with 4-aminodimethylaniline-3-sulphonic acid to give a greenish-blue wool dye. 1 : 3-*Di*-p-*anisylaminonaphthalene*-6 : 8-*disulphonic acid*, similarly prepared from disodium hydrogen α-naphthylamine-3 : 6 : 8-trisulphonate, condenses with nitrosodimethylaniline to give a pure blue wool dye. C. HOLLINS.

Manufacture of products of conversion of *peri*-cyanonaphthalenesulphonic acids [1 : 8-hydroxy- and -amino-naphthoic acids]. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 276,126, 28.7.26).—1-Cyanonaphthalene-8-sulphonic acid is hydrolysed by acids or alkalis to 8-sulpho-1-naphthamide, which is converted by acids into 8-sulpho-1-naphthoic acid or its inner anhydride, and then by alkaline fusion into 8-hydroxy-1-naphthoic acid. The 8-sulpho-1-naphthamide is converted by alkalis into 8-amino-1-naphthoic acid or its inner anhydride (naphthastyril). Thus sodium 1-cyano-8-naphthalenesulphonate, prepared from 1 : 8-naphthylaminesulphonic acid, is dissolved in concentrated sulphuric acid, diluted to 60% acid, and warmed at 70° to give 8-*sulpho*-1-*naphthoic acid* (*anhydride*, m.p. 151—152°), which, on alkaline fusion, yields 1 : 8-hydroxynaphthoic acid, m.p. 169°. Sodium 1-cyanonaphthalene-8-sulphonate is converted by alkaline fusion into naphthastyril, m.p. 180—181°, which by boiling with alkali gives 8-amino-1-naphthoic acid. Sodium 5-chloro-1-cyanonaphthalene-8-sulphonic acid similarly yields 6-chloronaphthastyril, m.p. 270°, hydrolysable to 5-chloro-8-amino-1-naphthoic acid. From 1-cyanonaphthalene-4 : 8-disulphonic acid 4-*hydroxy*-8-*sulpho*-1 *naphthamide* and 5-*hydroxynaphthastyril*, m.p. above 300° (*methyl ether*, m.p. 223°; *ethyl ether*, m.p. 200°), are obtained; the methyl ether is hydrolysed to 8-*amino*-4-*methoxy*-1-*naphthoic acid*. 1-Cyanonaphthalene-3 : 8-disulphonic acid gives 8-*amino*-3-*sulpho*-1-*naphthoic acid*, 4-*sulphonaphthastyril*, 4-*hydroxynaphthastyril*, 8-*amino*-3-*hydroxy*-1-*naphthoic acid*, 4-*alkoxynaphthastyrils*, and 8-*amino*-3-*alkoxy*-1-*naphthoic acids*. C. HOLLINS.

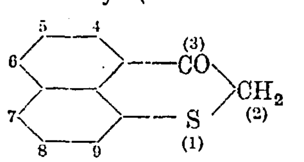
Manufacture of *ar*-tetrahydronaphthylamines or derivatives thereof. O. Y. IMRAY. From SOC. CHEM. IND. in BASLE (E.P. 276,571, 24.2.27).—*N*-Substituted α-or β-naphthylamines are reduced with hydrogen in presence of a nickel catalyst. The preparation of N-*ethyl*-ar-*tetrahydro*-α-*naphthylamine*, b.p. 139—142°/8 mm. (*acetyl* derivative, m.p. 58°, b.p. 167—168°/8 mm.), *ar*-α-acetamidotetraline, m.p. 159—160°, *ar*-β-acet-

amidotetraline, m.p. 109°, and N-*phenyl*-ar-*tetrahydro-α-naphthylamine*, m.p. 43·5°, b.p. 198°/8 mm., is described.
C. Hollins.

Manufacture of new products of the anthraquinone series [hexahydroarylaminoanthraquinones; acid wool dyes]. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 276,408, 13.5.26).—Hexahydroarylaminoanthraquinones are prepared by the usual methods from a *cyclo*hexylamine and a suitable anthraquinone derivative. 4-Bromo-1-aminoanthraquinone-2-sulphonic acid with *cyclo*hexylamine gives a clear blue wool *dye*, with 2-methyl*cyclo*hexylamine a greenish-blue. 4-*p*-*Toluidino*-1-cyclo*hexylaminoanthraquinone*, from 4-*p*-toluidino-1-methoxyanthraquinone and *cyclo*hexylamine, yields a greenish-blue wool *dye* on sulphonation. Quinizarin condenses with 2-amino*cyclo*hexanol to give 1 : 4-*di*-(2-*hydroxy*cyclo*hexylamino*)-*anthraquinone*, the sulphuric ester of which dyes wool a clear blue. The dyeings are fast to light. C. Hollins.

Production of chloro-derivatives of *N*-dihydro-1 : 2 : 1′ : 2′-anthraquinoneazine. O. M. Bishop, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,637,851, 2.8.27. Appl., 24.6.22).—The azine is treated in acetic acid or nitrobenzene suspension for varying periods at above 40° with dry chlorine to yield products which give in the vat greenish shades fast to chlorine. T. S. Wheeler.

Manufacture of new vat dyes containing sulphur [green indigoid dyes]. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 275,292, 9.4.26).—*peri*Naphthathioindoxyl (annexed formula) and its derivatives are



condensed with chlorides or α-anils of isatins, thioisatins, naphthisatins, or naphthathioisatins; the products may subsequently be halogenated. *peri*Naphthathioindoxyls are obtained from corresponding α-thionaphthols by condensation with chloroacetic acid, followed by cyclisation with chlorosulphonic acid; or 1 : 6-dibromo-β-naphthylamine is treated with sulphur chloride, and the resulting thiazthionium chloride is condensed with chloroacetic acid, acetylated, and finally cyclised to 5-*bromo*-9-*acetamido*peri*naphthathioindoxyl*. Green vat dyes result when a 9-amino*peri*naphthathioindoxyl is condensed with a thioisatin chloride or anil. *peri*Naphthathioindoxyl gives with 4 : 7-dichloroisatin chloride a blue vat dye, with 9-chloro-ββ-naphthathioisatin α-1-chloro-2-naphthylimine (E.P. 21,915 of 1912; B., 1913, 745) a blue-green, with the brominated 9-chloro-compound (*loc. cit.*) a yellow-green. 9-Acetamido*peri*naphthathioindoxyl with 4 : 5 : 6-trichloroisatin chloride gives a dark blue vat dye, or with β-naphthathioisatin a violet; both yield green shades after hydrolysis. 9-*Amino*peri*naphthathioindoxyl*, obtained from its acetyl derivative by hydrolysis with concentrated hydrochloric acid, condenses with 8-chloro-β-naphthathioisatin-2-*p*-dimethylaminoanil to form a yellowish-green vat dye. C. Hollins.

Manufacture of [thioindigoid] vat dyes. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 251,996, 5.5.26. Conv., 6.5.25).—Thioindigoid vat dyes of good fastness to light and kier-boiling are obtained from thioindoxyls derived from 4 : 5-dihalogeno-3-methylphenylthioglycollic acids or their 2-carboxy-, carbamido-, or cyano-derivatives. The thioindoxyls are oxidised to symmetrical thioindigos, or are condensed with chlorides or α-anils of thioisatins, or are converted into thioisatin derivatives and condensed with thioindoxyls. 4 : 5-Dichloro-2-cyano-3-methylphenylthioglycollic acid is converted by alkaline sodium sulphide at 80—85° into the 3-aminothionaphthene-2-carboxylic acid, which is hydrolysed to 5 : 6-dichloro-4-methylthioindoxyl. This is oxidised to the thioindigo (a blue-red vat dye, also obtained by the action of chlorosulphonic acid on 4 : 5-dichloro-3-methylphenylthioglycollic acid), or converted by means of nitrosodimethylaniline into the corresponding thioisatin α-*p*-dimethylaminoanil, which is condensed with 9-bromo-ββ-naphthathioindoxyl to give a violet dye.
C. Hollins.

Manufacture of nitrobenzene. J. W. Livingston and L. P. Kyrides (U.S.P. 1,638,045, 9.8.27. Appl., 27.11.22).—A mixture of sodium nitrate, 95% sulphuric acid, and benzene is added at 80—110° during 3 hrs. to a mixture of 81% sulphuric acid, benzene, and nitrobenzene, the whole being then heated at 110° for 1 hr. to give nitrobenzene in theoretical yield.
T. S. Wheeler.

Nitration process. M. Battegay (E.P. 262,097, 19.11.26. Conv., 24.11.25).—Aromatic compounds are nitrated with nitrogen tetroxide in presence of concentrated sulphuric acid, the products being the nitro-derivative and nitrososulphonic acid (nitrosylsulphuric acid). Benzene (100 pts.) below 24° gives 40 pts. of pure nitrobenzene and 71 pts. of recovered benzene. Toluene (100 pts.) at 22° gives 11 pts. of nitrotoluenes (65% *o*-, 25% *p*-). Chlorobenzene (100 pts.) at 10° gives 51 pts. of chloronitrobenzenes (mainly *para*).
C. Hollins.

Manufacture of quinoline derivatives. British Dyestuffs Corp., Ltd., and M. Wyler (E.P. 276,156, 6.9.26).—Quinaldine bases are isolated from the mixture of products (obtained, *e.g.*, in the Döbner–Miller reaction) in the form of zinc chloride double salts. When these double salts are distilled, very pure hydrochlorides of the bases pass over between 250° and 300°. Quinaldine zinc chloride, m.p. about 240°, in this way gives quinaldine hydrochloride, m.p. 211°; o-*toluquinaldine* [2 : 8-*dimethylquinoline*] *zinc chloride*, m.p. 273°, gives *o*-toluquinaldine hydrochloride, m.p. 227°. The free bases, liberated from the zinc double salts or hydrochlorides, have b.p. 247—250° and 256° (m.p. 27°), respectively.
C. Hollins.

Manufacture of *cyclo*hexylamines. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 261,764, 18.11.26. Conv., 19.11.25).—*cyclo*Hexyl halides are made to react with primary or secondary arylamines, preferably in presence of acid-binding agents or catalysts, *e.g.*, copper. Bromo*cyclo*hexane with aniline yields cyclo*hexylaniline*, b.p. 142°/15 mm. cyclo*Hexylethylaniline* boils at 148—152°/15 mm., cyclo*hexylmethylaniline* at 145—148°/15 mm., (2-*methyl*cyclo*hexyl*)*aniline* at 146—152°/15 mm.; 1-cyclo*hexylaminoanthraquinone* is also described. C. Hollins.

Production of indophenol compounds. W. A. Manss, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,637,868, 2.8.27. Appl., 14.12.23).—The process of E.P. 9689 of 1909 (B., 1910, 481) is modified in that the condensation of *N*-ethylcarbazole and *p*-nitrosophenol is rapidly effected at below — 13°, the product being converted immediately into the stable leuco-compound by treatment with sodium sulphide. T. S. Wheeler.

Manufacture of salts of o-aminophenylpropionic acid, its homologues, substitution products, and analogues. I. G. Farbenind. A.-G. (F.P. 613,596, 27.3.26. Conv., 6.4.25).—Compounds of the dihydrocarbostyril type are heated with aqueous alkalis, *e.g.* with barium hydroxide solution at 150°. Dihydrocarbostyril gives sodium β-*o*-aminophenylpropionate; 8-methyldihydrocarbostyril, prepared from β-chloropropion-*o*-toluidide and aluminium chloride (G.P. 415,096; B., 1925, 865), gives sodium β-(2-amino-*m*-tolyl)propionate; 4-methyldihydrocarbostyril, from β-chlorobutyranilide, gives sodium β-*o*-aminophenylbutyrate; and from 2 : 9-diketo-1 : 2 : 3 : 4 : 7 : 8 : 9 : 10-octahydro-*o*-phenanthroline there is obtained a salt of 5 : 6-diamino-1 : 4-phenylene-ββ-dipropionic acid [5 : 6-diamino-1 : 4-di-(β-carboxyethyl)benzene]. C. Hollins.

Cyclic hydrocarbons and derivatives (E.P. 253,911).—See XX.

Sulphonic acids (E.P. 269,155).—See XX.

Oxidising organic compounds (E.P. 259,930).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Colloid chemistry of "viscose" solutions. V. Viscosity minima of "viscose" solutions and their alteration with time. VI. Surface tension of "viscose" solutions. T. Mukoyama (Kolloid-Z., 1927, **42**, 350—353, 353—355).—V. The viscosity of a solution of viscose undergoes a change with time, falling during the first few days to a minimum value, after which it rises. With increasing alkali content, the viscosity of the viscose is lowered and the position of the minimum is displaced in the sense of a retardation of ageing. Dilution of the viscose solution with water causes the minimum to appear earlier; the interval after which the minimum viscosity appears is directly proportional to the concentration of the viscose solution. The viscosity is reduced by mechanical agitation, and the minimum point is more retarded the more severe the agitation. The existence of a viscosity minimum is due to the simultaneous effects of gelatinisation and desolvation. VI. Contrary to the behaviour of the viscosity, the surface tension of solutions of viscose does not change with time over a period of ten days. Measurements carried out by the drop-number method showed a decreased surface tension for slowly formed drops, suggesting membrane formation or adsorption at the surface. E. S. Hedges.

Bleaching of wood cellulose. IV. Influence of metals and salts on hypochlorites and the bleaching process. H. Wenzl (Papier-Fabr., 1927, **25**, Fest- u. Ausland-heft, 76—85. Cf. B., 1924, 89; 1926, 267).—The catalytic decomposition of hypochlorites by metals, *e.g.*, copper and iron, with evolution of oxygen, is well known in the bleaching industry. Various metals and alloys differ widely in their catalytic activity, lead, tin, and bronze being nearly inactive, whilst nickel, cobalt, and manganese decompose the hypochlorites with extraordinary rapidity. As the concentration of the hypochlorite solution is increased the velocity of the decomposition is reduced at first and then becomes constant. Increase of concentration of hydroxyl ions has also a retarding influence. Rise of temperature accelerates the reaction. Although in certain circumstances the catalytic evolution of active oxygen in the presence of metals may cause profound damage to the fibre, it is nevertheless possible, by regulating the rate of evolution of oxygen to suit the rate of consumption of oxygen by the incrusting matters, to accelerate the useful bleaching process without wasteful destruction of hypochlorite or damage to the cellulose. The action of the metal on the hypochlorite is complicated by the formation of coloured hydroxides which appear to act as intermediaries. The acceleration of the bleaching process is utilised industrially by suspending discs of nickel or other suitable metal or alloy in the circulating pulp in the presence of bleach liquor and by regulating the rate of reaction by increasing the surface of metal exposed, or by raising the temperature, or *vice versa*. Analyses of pulps bleached with the assistance of an accelerator showed higher α-cellulose values and lower copper values than those bleached in the ordinary way. J. F. Briggs.

Viscosity of nitrocellulose. O. Merz (Farben-Ztg., 1927, **32**, 2768—2771, 2886—2887).—Published literature on the viscosity of nitrocellulose solutions and its determination is summarised. For exact measurements the Engler viscosimeter is recommended, reduction in the standard efflux volume and use of a wider aperture (5 mm. diam.) being necessary in the case of highly viscous solutions. Special consideration is given to the influence of the alcohol content of moist nitrocellulose on the viscosity of solutions of the latter, removal of the alcohol not being in accordance with practical usage. S. S. Woolf.

Examination of decayed papier maché fire buckets. A. C. Thaysen and H. J. Bunker (J.S.C.I., 1927, **46**, 382 T).—It was found that papier maché fire buckets became soft and collapsed after two or three months' use. This was shown to be due to micro-organisms present in the paste or glue used in making the buckets. The addition of an antiseptic, such as 1% of phenol, to the paste extended the life of the buckets almost indefinitely. Such treatment could be employed for any papier maché articles intended to hold water for prolonged periods.

Patents.

Dressing of textile materials. J. F. King (E.P. 275,891, 14.3.27).—Weighting and filling materials are added to the kier boiling bath. *E.g.*, grey goods are entered into a kier solution containing 300 lb. of sodium perborate, 30 lb. of trisodium phosphate crystals, and 25 lb. of aluminium sulphate, to which have been added 50 lb. of corn starch, 75 lb. of magnesium sulphate, and 20 lb. of denatured alcohol, and boiled under pressure for 4—6 hrs. C. Hollins.

Manufacture of an [insulating] composition. H. FRIEDLANDER (E.P. 275,795, 18.8.26).—Textile or asbestos fibres in the form of coherent, homogeneous felts, containing if required a proportion of cellulosic material such as wood pulp, are impregnated with artificial resins under the action of vacuum or pressure, dried, and submitted to pressure while hot.

B. P. RIDGE.

Process for chemically varying vegetable and artificial fibre. HEBERLEIN & Co., A.-G. (E.P. 261,793, 22.11.26. Conv., 21.11.25. Addn. to E.P. 255,453; B., 1927, 103).—The process of the prior patent, namely treatment of vegetable fibre with a phosphorus halide in presence of alcoholic alkali, is preceded or followed by a separate treatment with alkali, and is extended also to artificial silk of all kinds. C. HOLLINS.

Production of cellulose acetate. SOC. CHIM. DES USINES DU RHÔNE (F.P. 615,879, 10.5.26).—Apparatus is used provided with stirrers adapted to the different conditions of the acetylating mixture in the separate phases. Thus a vertical shaft to which various stirring arms are attached passes through a tube into which the acetylating mixture is fed from above. In the upper part of the apparatus the shaft carries hooks in order to mix the softened fibres through one another; below this powerful stirring arms are fixed, whilst in the lower part discs are used to mix the gummy mass. The finished mixture is drawn off from the lower end of the tube. B. P. RIDGE.

Purification, decomposition, and stabilisation of cellulose esters, cellulose ethers, and artificial materials produced therefrom. WOLFF & Co., and H. SCHULZ (G.P. 440,844, 5.12.25).—The material is cooked with water under atmospheric pressure, steamed, or subjected to the action of a vacuum with heating or at the ordinary temperature. By this treatment the decomposition of the cellulose derivatives in the subsequent cooking is avoided. B. P. RIDGE.

Manufacture of textile threads from viscose solutions. R. FRANKL (Swiss P. 117,136, 1.4.25. Conv., 10.4.24).—The thread issuing from the spinning bath which, besides sodium hydrogen sulphate, contains free sulphuric acid, is taken up by a spinning centrifuge, a permanent stretching of the thread being thereby produced. B. P. RIDGE.

Process of bleaching cellulosic material. G. A. RICHTER and M. O. SCHUR, Assrs. to BROWN Co. (U.S.P. 1,639,704, 23.8.27. Appl., 27.5.24).—Bleached wood pulp rich in α-cellulose is made by adding alkali to the bleaching liquor in sufficient quantity to maintain a distinctly alkaline condition throughout the bleaching operation. W. G. CAREY.

Reduction of the swelling capacity of artificial products from cellulose and its derivatives. F. HEBLER (Austr. P. 105,353, 27.3.22).—The products are heated to above 115° in the presence of formaldehyde. Before the heating, ammonium salts, bases, or basic salts and/or salts of organic acids may be incorporated with or deposited on them. B. P. RIDGE.

Dissolving out lignin and similar incrusting substances from wood and other plant fibre materials, such as cellulose. DRESDENER CHROMO- & KUNSTDRUCK-PAPIERFABR. KRAUSE UND BAUMANN A.-G., and H. SCHWALBE (G.P. 441,392, 10.8.24).—The material, cut up in the usual manner, is surrounded by an atmosphere of gas (carbon dioxide, hydrogen chloride, sulphur dioxide) and shaken with concentrated sulphuric acid, whereby a rapid dissolution of the non-incrusting substances occurs. The solution is readily filtered, the incrusting materials remaining behind as crystalline, light brown particles. B. P. RIDGE.

Artificial silk spinning machine. J. L. RUSHTON (E.P. 276,748, 3.6.26).

Manufacture of centrifugal pots for use in spinning artificial silk. F. SCARPA, and ANC. ETABL. J. JUTHY (E.P. 271,892, 26.5.27. Conv., 26.5.26).

Method and means for washing and conditioning artificial silk wound on bobbins etc. BRYSILKA, LTD., and F. W. SCHUBERT (E.P. 274,928, 29.3.26).

Recovery of caustic soda from residual lyes (E.P. 267,536).—See VII.

Sulphite liquors (U.S.P. 1,637,514—5 and 1,637,353, and G.P. 441,178).—See VII.

Metallising textiles etc. (E.P. 252,710).—See XI.

Hair felt (G.P. 440,401 and Austr. P. 103,897).—See XV.

Stabilisation of hydrocarbons used in cleaning etc. (F.P. 615,800).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Fastness to light of dyestuffs on woollen and worsted fabrics. I. Comparison of the fading of dyestuffs in tropical and in English sunlight and by artificial light. S. G. BARKER and H. R. HIRST. **II. Atmospheric humidity and the fading of dyestuffs.** J. J. HEDGES. **III. Relation between time of exposure and loss of colour due to fading.** S. G. BARKER, H. R. HIRST, and P. N. LAMBERT. **IV. Relation between initial depth of shade and loss of colour due to fading.** S. G. BARKER, H. R. HIRST, and P. N. LAMBERT (J. Soc. Dyers and Col., 1927, **43**, 254—261, 261—262, 263—264, 264—266).—I. The fading of woollen and worsted patterns dyed with about 60 acid, direct, vat, and mordant dyes when exposed to tropical (India and Ceylon) and English sunlight, and to light from a Fadeometer was determined by means of a Lovibond tintometer, by direct visual comparison, and by comparison while illuminated by ultra-violet light. It was shown that fading could be classified under such headings as simple loss of colour (the hue of the material being unaffected), actual change of colour hue, and loss of brightness without loss of colour. The results obtainable by comparison during exposure to ultra-violet light of the faded material are dependent on the fact that dyed wool exhibits no fluorescence during such exposure, but becomes fluorescent under similar conditions after exposure to sunlight with consequent fading. By comparison of the fluorescent properties of the faded patterns it was possible to distinguish which patterns had been faded in England, in the tropics, or by a Fadeometer. The fading results obtained by means of a Fadeometer did

not agree with those obtained by exposure of the patterns to direct sunlight, and actually less fading occurred in the tropics than in England. These discrepancies are largely due to differences in the humidity conditions of the atmosphere surrounding the patterns during exposure.

II. The amount of fading produced by exposure to ultra-violet light (from a "Hanovia" mercury vapour lamp) of wool patterns dyed with Ponceau RG, Acid Green G, and Cyanine B under various determined conditions of humidity indicates that the loss of colour during fading increases with increasing humidity of the atmosphere surrounding the dyed patterns. *E.g.*, the losses of the predominant colours in patterns dyed with Ponceau RG, Acid Green G, and Cyanine B maintained at 52 and 100%, 33 and 100%, and 24 and 100% relative humidity were 1·5 and 4·9%, 11·2 and 21·7%, and 23·8 and 41·0%, respectively. Humidity must be taken into account as an important factor in tests of fastness to light of dyed materials.

III. The progressive loss of the predominant colour in worsted fabrics dyed with Indigo Carmine X, Wool Green S, Neolan Green B, Acid Violet 6B, Magenta, Ponceau RG, and Chlorazol Blue B produced by fading due to prolonged exposure to light from a Fadeometer may be expressed by the equation $T = \alpha L^n$, where T is the time of exposure in hours, L is the percentage loss of predominant colour, α is a constant dependent on the dye, and $n = 1 \cdot 9$ or 2. The actual and calculated colour losses differ slightly during the early stages of fading.

IV. Measurement of the proportional losses of the predominant colour in worsted fabrics dyed to different depths of shade with each of 15 acid, mordant, and direct dyes produced by exposure to sunlight and light from a Fadeometer showed that the loss of colour during fading was independent of the depth of shade.

A. J. HALL.

Identity of digallic acid with tannin as mordant for basic dyes. P. P. VIKTOROV and Z. N. SIRKIN (J. Russ. Phys. Chem. Soc., 1926, **58**, 1351—1361).—See B., 1927, 746.

Bleaching of wood cellulose. WENZL.—See V.

PATENTS.

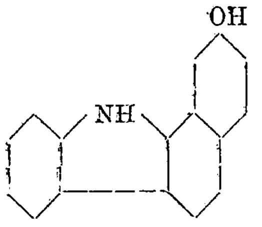
Apparatus for bleaching textiles by means of oxygen or ozone baths. R. MOHR (G.P. 441,269, 28.11.24. Addn. to G.P. 421,906; B., 1926, 403).—An apparatus for carrying out the process described in the chief patent comprises two bleaching vats which may be used alone or in series with one another; each is coupled with a special pressure pump and with a common pipe leading to the reservoir holding the overflow of the pressure liquid by means of a regulating valve so arranged as to supply pressure to either vat singly, or to the whole apparatus. Below the vats is a pressure tube joining the vats to the circulating pump, and provided with valves and connexions to the pressure vessel and the upper stopcocks of both bleaching vats.

A. R. POWELL.

Dyeing artificial [regenerated cellulose] silk. BRITISH DYESTUFFS CORP., LTD., J. BADDILEY, P. CHORLEY, and C. BUTLER (E.P. 276,450, 4.6.26).—Level shades on viscose silk are obtained by using secondary disazo dyes of the type: aminosalicylic acid or homologues → usual middle components → sulphonated 1 : 8-dihydroxynaphthalene or 1 : 8-aminonaphthol. The shades obtained are grey-blue, blue, and blue-black; thus, 5-aminosalicylic acid → α-naphthylamine → 2S-acid (coupled alkaline) gives a blue-black; 5-amino-*p*-cresotic acid → α-naphthylamine → 1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid gives a blue.

C. HOLLINS.

Dyeing and printing with ice colours. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 275,326, 4.5.26).—The fabric is padded with a hydroxylated naphtha- or dinaphtha-carbazole and developed with an unsulphonated diazo compound. The carbazoles are obtained by the Bucherer reaction from arylhydrazines and naphthol- or naphthylamine-sulphonic acids, followed by alkaline fusion. 2-Hydroxy-α-naphthacarbazole (annexed formula), coupled on the fibre with diazotised *m*-nitroaniline, gives a brown-red; brominated 2-hydroxy-α-naphthacarbazole with diazotised 4-nitro-*o*-anisidine, a brown-black; 3-hydroxy-α-naphthacarbazole with diazotised 4-nitro-*o*-anisidine, a violet-black; 2-hydroxy-9-methyl-α-naphthacarbazole with diazotised 4-nitro-*o*-toluidine, a bordeaux. The dyeings are fast to boiling, chlorine, light, and bowking.



C. HOLLINS.

Dyeing of effect threads. G. B. ELLIS. From CHEMICAL WORKS, FORMERLY SANDOZ (E.P. 276,100, 7.6.26).—Immunised effect threads (cf. E.P. 195,619; B., 1924, 128) are dyed directly, or by development, with substantially insoluble dyes of all classes in presence of a dispersing or solubilising agent other than the higher fatty acids, or sulphonic derivative thereof, mentioned in E.P. 246,609 (B., 1926, 270). The non-immunised material may be dyed independently with direct dyes in the same bath.

C. HOLLINS.

Dyeing of cellulose ester artificial silk. BRITISH ALIZARINE CO., LTD., W. H. DAWSON, C. W. SOUTAR, and R. J. WOOD (E.P. 275,752, 27.5.26).—Yellow to orange shades are obtained by dyeing acetate silk with mono-, di-, or tetra-nitroacridines.

C. HOLLINS.

Recovery of hemicellulose-free sodium hydroxide mercerisation liquor. DEUTS. ZELLSTOFF-TEXTILWERKE G.M.B.H., Assees. of K. LEUCHS (G.P. 441,424, 19.12.22).—The liquor is saturated with carbon dioxide, whereby most of the hemicellulose is precipitated, the sodium carbonate or bicarbonate crystallised out and causticised with calcium hydroxide.

B. P. RIDGE.

Treatment [dyeing] of cellulose derivatives. G. H. ELLIS, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,641,965, 13.9.27. Appl., 27.9.24. Conv., 8.10.23).—See E.P. 227,183; B., 1925, 204.

Production of ice colours on cotton. I. G. FARBENIND. A.-G., Assees. of K. JELLINCK and W. CHRIST (U.S.P. 1,638,121, 9.8.27. Appl., 15.1.25. Conv., 25.2.24).—See E.P. 230,029; B., 1925, 878.

Rollers for calendering machines. I. G. FARBENIND. A.-G. (E.P. 264,179, 10.1.27. Conv., 8.1.26).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Rate of absorption of hydrogen chloride in aqueous solutions. H. O. Askew (J.S.C.I., 1927, **46**, 386—390 T).—The rate of absorption of hydrogen chloride from air bubbles in aqueous alkali solutions, with or without the addition of capillary-active substances, has been studied. By varying the rate of passage of the gas through the solutions it was found that the final partial pressure of acid vapour in the escaping gas was directly proportional to the time of formation of the bubble; hence, extrapolation could be made to zero time of formation for determination of the partial pressure to be used in calculating the over-all coefficient of absorption. From the over-all coefficient k the separate coefficients k_w, k_a, k_c, for the air-liquid, the alkali, and the colloid film respectively, have been calculated from the equation $1/k = 1/k_w + 1/k_a + 1/k_c$. The rate k_a increased rapidly with decrease in the alkali concentration. Addition of capillary-active substances greatly decreased the rate of absorption.

Simple mercury cathode for arsenic determinations. F. S. Aumonier (J.S.C.I., 1927, **46**, 341—345 T).—A mercury cathode cell for the electrolytic micro-determination of quinquevalent arsenic is described. The cell is made from materials easily obtainable. The arsenic produced by electrolysis is decomposed by heat from a small electric furnace in series with the cell, and the arsenic is deposited in a tube of standard dimensions. "Poisoning" of the cathode is avoided by preliminary wet combustion, and hydrochloric acid distillation applied to samples of food. The method described has been found more convenient and certain than the electrolytic method in which a platinum cathode is used.

Manufacture of bromides and iodides. F. Chemnitius (Chem.-Ztg., 1927, **51**, 587—588, 608—609).—Potassium bromide is usually manufactured by the interaction of potassium carbonate and a solution of iron bromide, Fe_3Br_8, the latter being prepared by running bromine on to iron under water. Precipitated iron hydroxide and carbonate are removed by filtration, and the solution is concentrated by evaporation. The mother-liquor is evaporated to dryness and the residue roasted with charcoal to reduce potassium bromate. Potassium iodide, sodium bromide, and sodium iodide are manufactured by analogous processes, except that potassium iodate is preferably converted into iodide by means of barium sulphide. Ammonium bromide is prepared by the action of bromine on ammonia, and ammonium iodide from iodine and ammonium sulphide.
W. T. K. Braunholtz.

Factors influencing the loss of iodine from iodised salt. A. H. Johnson and B. L. Herrington (J. Agric. Res., 1927, **35**, 167—183).—The loss of iodine from iodised salt (1 oz. of potassium iodide per 100 lb. of salt) varied with the humidity of the atmosphere, being least at 50% relative humidity. Exposure to heat and sunlight increased the loss of iodine. These effects are prominent in neutral or acid salts, but the addition of sodium bicarbonate to produce an alkaline reaction practically eliminated all losses under any conditions of storage. The loss may also be obviated by iodising with potassium iodate. The rate of liberation of iodine from neutral salt-potassium iodide mixtures varied with the proportion of the latter used. A. G. Pollard.

Determination of iodide in mixtures of halides. H. Baines (J.S.C.I., 1927, **46**, 381—382 T).—The method described by Chick (B., 1927, 651) for determining iodide in mixtures of halides was independently developed for determination of iodide in photographic products. The small quantities obtainable and the ratio of iodide to other halides necessitated certain precautions.

Tower absorption coefficients. Cantelo and others.—See I.

Perchloric acid as oxidising agent. Kahane.—See XIV.

Patents.

Platinum contact mass for use in the conversion of sulphur dioxide into sulphur trioxide. T. von Artner (G.P. 440,338, 16.2.26).—To prevent useless deposition of platinum inside the mass of the carrier the latter (*e.g.*, anhydrous magnesium sulphate) is first impregnated with a volatile organic liquid, *e.g.*, alcohol, then dipped in an alcoholic solution of a platinum compound. The mass is then quickly dried and heated to decompose the platinum compound, leaving finely-divided platinum on the surface of the carrier.
A. R. Powell.

Absorption of oxides of nitrogen. T. Schloesing (Addn. No. 31,253 and 31,301, [A] 11.4.25, [B] 5.11.25, to F.P. 609,264; B., 1927, 252).—(A) A neutral or acid solution formed during the absorption process, *e.g.*, calcium nitrate solution, is used as the absorbing liquid. (B) The gases are absorbed at 40—80° by an insoluble or slightly soluble base, such as calcium carbonate, worked up to a thin paste with water. L. A. Coles.

Production of nitric acid. Metallbank. u. Metallurgische Ges. A.-G., and Deutsche Sprengstoff-A.-G., Assees. of W. Melzer and W. von Boltenstern (G.P. 440,657, 1.5.25).—In the electrical precipitation of nitric acid from a mixture of air, nitrous gases, dilute acid mist, and steam, the quantity of steam is so regulated that concentrated acid is precipitated. L. A. Coles.

Method of stabilising hydrocyanic acid. Deutsche Gold- u. Silber-Scheideanstalt, vorm. Roessler (E.P. 254,747, 3.7.26. Conv., 3.7.25).—Hydrogen cyanide solutions (94—98%) are stabilised by addition of small amounts of substances, *e.g.*, ethyl sulphate, aluminium chloride, chloropicrin, methyl chloroformate, ethyl bromoacetate, which by hydrolysis liberate acid ions. Other such stabilisers are halides of antimony, aluminium, lead, tin, arsenic, etc., zinc and iron salts, alkyl halides, sulphates, borates, oxalates, organic acid chlorides, chloroform, carbon tetrachloride, trichloroethylene, etc.
C. Hollins.

Recovery of caustic soda from the residual lyes after the manufacture of artificial silk. Appareils et Evaporateurs Kestner (E.P. 267,536, 10.3.27. Conv., 12.3.26).—The cold lye is subjected to a current of carbon dioxide, and after filtration the resulting soda crystals are dissolved and treated with lime to remove the carbon dioxide. W. G. Carey.

Process for the direct synthesis of ammonia.

F. C. Reed (U.S.P. 1,639,272, 16.8.27. Appl., 21.1.26).—A mixture of nitrogen and hydrogen under pressures of up to 400 atm. is passed at temperatures of up to 500° through a series of catalysts, the ammonia contained in the gases leaving each catalyst being removed at a lower temperature, and the gases being continuously re-circulated after the addition of further quantities of nitrogen and hydrogen. The gases pass from an outer to an inner catalytic chamber, the conditions being so adjusted that the temperature of the inner chamber is 50—150° above that of the outer chamber.

C. O. Harvey.

Synthesis of ammonia. L. Tocco and M. Landi (Addn. No. 31,202, 27.11.25, to F.P. 607,059; B., 1927, 218).—Barium carbide is treated with nitrogen to obtain barium cyanide which, by the action of hydrogen, is reconverted into carbide with the formation of ammonia.

A. R. Powell.

Production of caustic alkali etc. L. Bradley and E. P. McKeefe (U.S.P. 1,637,514, 2.8.27. Appl., 2.3.21. Renewed 8.11.26).—An alkali sulphate is treated in dilute acid solution with a suitable insoluble calcium salt, *e.g.*, calcium phosphate or sulphite, to yield calcium sulphate and an alkali salt: the latter can be causticised with lime. T. S. Wheeler.

Treatment of residual [sulphite] liquors. L. Bradley and E. P. McKeefe, Assrs. to Bradley-McKeefe Corp. (U.S.P. 1,637,515, 2.8.27. Appl., 7.2.22. Renewed 15.10.26).—Waste sodium sulphite liquor is concentrated to separate sodium sulphite and acidified to precipitate organic matter, being then treated with sulphur dioxide to convert sodium compounds into sodium sulphite, and with lime partially to causticise the latter. Alternatively, the alkaline liquor is treated with calcium sulphate to give sodium sulphate and insoluble calcium sulphite. The solution is evaporated, and the residue incinerated. The sodium sulphate remaining is heated with calcium sulphite in acid solution (cf. preceding abstract).

T. S. Wheeler.

Manufacture of sulphite cooking liquor. G. A. Richter, Assr. to Brown Co. (U.S.P. 1,637,353, 2.8.27. Appl., 28.9.21).—Gas from a sulphur burner, with about 16% of sulphur dioxide, is passed in counter-current to a stream of water through towers containing calcium carbonate, the sulphite liquor obtained being treated with gas blown from a sulphite pulp digester to increase the free sulphur dioxide content. T. S. Wheeler.

Production of sulphite liquor. A. Schneider (G.P. 441,178, 7.1.25).—Milk of lime is treated under pressure with gases containing sulphur dioxide, which are either drawn straight from the producer or purified from gases or vapours other than sulphur dioxide before being absorbed. In the latter case the gases not absorbed, which are withdrawn from the process, are again conducted partially over the apparatus for the production of sulphur dioxide. For the absorption, rotating pressure containers filled with limestone are also used.

B. P. Ridge.

Manufacture of sodium sulphites and boric acid. H. Blumenberg, jun., Assr. to Stockholders Syndicate (U.S.P. 1,642,535, 13.9.27. Appl., 4.9.24).—A solution of borax is treated with sulphur dioxide until the reaction forming normal sodium sulphite and boric acid is seen to be complete by the formation of a cloudy precipitate of sodium bisulphite, the precipitated boric acid is separated, and the remaining mixture is treated with sulphur dioxide to form sodium bisulphite.

W. G. Carey.

Production of sodium hydrosulphide. B. Reinhardt (G.P. 435,527, 22.9.25).—Barium hydrosulphide solution, formed by treating an aqueous suspension of barium sulphide with hydrogen sulphide, is converted wholly or partially into sodium hydrosulphide solution by treatment with sodium chloride, hydrogen sulphide being expelled from the residues and used again in the process. L. A. Coles.

Production of ammonium chloride from ammonia-soda mother-liquors. I. G. Farbenind. A.-G., Assees. of R. Griessbach and J. Giesen (G.P. 440,918, 12.5.23).—Nitrogen obtained by the fractional distillation of liquid air is passed directly into the liquor to cool it sufficiently to cause crystallisation of the ammonium chloride. L. A. Coles.

Briquetting sublimed ammonium chloride. Continentale A.-G. für Chemie (G.P. 440,704, 28.1.26).—The addition of 2—3% of crystalline ammonium chloride to powdered sublimed ammonium chloride renders it capable of compression into briquettes. L. A. Coles.

Dissolving aluminiferous raw materials by means of sulphuric acid or acid sulphates. R. Jacobsson (E.P. 269,174, 4.4.27. Conv., 6.4.26).—Bauxite, clays, etc. as silt are supplied under pressure and at a regulated speed to an autoclave charged with sulphuric acid or an acid sulphate, or the materials are mixed outside the autoclave and the mixture is supplied to the vessel at a regulated speed. The reaction temperature is 185° and the products are continuously or intermittently drawn off, fresh raw material being added at the same time. The solution of aluminium sulphate is separated from precipitated silica at a temperature of at least 120°. W. G. Carey.

Production of chromium compounds. L. Mellersh-Jackson. From Mathieson Alkali Works (E.P. 276,120, 20.7.26).—A non-oxidised mixture for alloy of chromium and iron, *e.g.*, ferrochromium, is finely ground and chlorinated by agitation in an atmosphere or counter-current of chlorine under non-oxidising conditions and not lower than 350° to volatilise the ferric chloride. The temperature is then raised above 650° and, after removal of the dissociated chlorine, the chromium chloride is delivered into a tall vertical column, so that it is cooled out of contact with air, the temperature of the material at the base being below 250°. Inert heat-storing material is mixed with the ferrochromium to assist in controlling and equalising the temperature.

W. G. Carey.

Production of magnesium chromate. I. G. Farbenind. A.-G. (F.P. 617,235, 9.6.26. Conv., 22.6.25).—The compound is produced by the double decomposition of an alkali or alkaline-earth chromate and a magnesium salt of an acid yielding an alkali or alkaline-earth salt less soluble than magnesium chromate.

L. A. Coles.

Method of transforming titanium dioxide or rutile. S. J. Lubowsky, Assr. to Metal & Thermit Corp. (U.S.P. 1,640,952, 30.8.27. Appl., 5.12.25).—Titanium dioxide or rutile is rendered soluble by calcination with magnesium oxide to approximately No. 18 Seger cone. W. G. Carey.

Production of molybdates. A. Kissock (E.P. 276,155, 4.9.26).—Finely-divided molybdenum trioxide, obtained by roasting molybdenite etc., and powdered slaked lime are intimately mixed, sufficient water is added to work the material into a pasty condition and to form calcium molybdate, and the mixture dried (cf. B., 1926, 792). W. G. Carey.

Distillation or concentration of hydrogen peroxide. Chem. Fabr. Coswig-Anhalt G.m.b.H., and E. von Drathen (G.P. 441,259, 14.4.26).—The liquid is distilled in apparatus constructed of quartz, porcelain, or stone, the walls having insulated tantalum wire or gauze attached to them. L. A. Coles.

Production of pure carbon dioxide, together with valuable by-products. P. Kircheisen (G.P. 441,224, 1.6.26).—Barium chloride prepared from barium sulphide is converted into barium carbonate and ammonium chloride by treatment with ammonia and carbon dioxide, and the carbonate is decomposed by treatment with sulphuric acid mixed with a little hydrochloric acid. L. A. Coles.

Controlling the production of carbon dioxide. E. W. Geere (E.P. 276,146, 4.11.26).—Acid phosphates, acid sulphates, tartaric or citric acid, etc., used to act on carbonates or bicarbonates, are treated with waxes, hydrogenated oils, or fats at the m.p. of the latter, and to the cooled homogeneous mixture is added an amount of the untreated acid medium in a suitable mixing mill. W. G. Carey.

Production of phosphorus mist. Chem. Fabr. Dr. H. Stoltzenberg (G.P. 440,843, 29.4.26).—Phosphorus liquefied by means of steam or other heating agents is injected through atomisers into the air by steam or other gas under pressure. L. A. Coles.

Granular caustic alkalis and method and apparatus for producing same. R. E. Wiley and C. E. Mensing (E.P. 275,044, 11.10.26).—See U.S.P. 1,601,897—8; B., 1927, 11.

Manufacture of sodium thiosulphate. F. W. Sperr, jun., and D. L. Jacobson, Assrs. to Koppers Co. (U.S.P. 1,639,905, 23.8.27. Appl., 4.4.21).—See E.P. 209,169; B., 1924, 164.

Forming metal phosphides and mixtures containing them. W. Koehler (E.P. 276,112, 25.6.26).—See U.S.P. 1,599,618; B., 1927, 11.

Atomiser for corrosive liquids (E.P. 271,035).—See I.

Ammonium nitrate and nitrite from combustion gases (G.P. 440,622).—See II.

Mixed manure (E.P. 275,843).—See XVI.

VIII.—GLASS; CERAMICS.

Patents.

Refractory brick. J. Schaefer (Can. P. 263,549, 22.10.25).—Magnesite, as free as possible from fluxing constituents, is burnt to magnesia at 1200—1400° and the product finely ground and mixed with a quantity of the finely-ground raw magnesite. The mixture is briquetted, sintered at 1600—1700°, and broken into small granules. These are mixed with ground, dead-burnt magnesite, and the product is formed into bricks which are fired at 1400—1500°. A. R. Powell.

Tunnel ovens or tunnel kilns for use in the manufacture of tiles, pottery, and other ware etc. J. Marlow (E.P. 275,048, 18.10.26).

[Machine for] cleaning clay. C. L. Crabb (E.P. 252,379, 19.5.26).

IX.—BUILDING MATERIALS.

Comparison of the economics of the boiler and rotary furnace processes for burning quick-setting plaster of Paris. B. Sägebarth (Chem.-Ztg., 1927, **51**, 588—589).—Disadvantages of the boiler process include preliminary fine grinding of the gypsum, discontinuous charging and discharging operations, indirect system of heating, and dependence on personal reliability of the operatives. With a rotary furnace, coarsely crushed gypsum (walnut size) passes continuously in the same direction as the heating gas, mixing and constant agitation of the material being aided by crossed baffles inside the furnace. Fine grinding is carried out after passage of the material through the furnace, this requiring less power than the fine grinding of the raw material. A saving in fuel of about 30% over the boiler process is claimed. W. T. K. Braunholtz.

Moisture content of wood. Stamm.—See XI.

Protective coatings for wood. Browne.—See XIII.

Patents.

Manufacture of lime, gypsum, calcareous and/or aluminiferous cements, etc., and/or calcination of ores etc., and kilns therefor. G. Martin (E.P. 276,066, 17.5.26).—A shaft kiln for roasting, calcining, and/or sintering ores etc. in powdered form consists of a relatively narrow or elliptical decarbonating or preheating zone with lateral openings through which the raw materials are introduced, a narrower sintering zone underneath arranged asymetrically with the decarbonating zone, so that hot gases introduced at the lower end of the sintering zone heat the wall of the decarbonating zone opposite that through which the raw materials are introduced, and a cooling shaft forming an extension of the sintering shaft below the furnace level through which air for combustion is introduced and preheated. The zones are so proportioned that the speed of the hot gases through the sintering zone is at least four times as great as when ascending the decarbonating zone, and a rotational movement is imparted to the particles in the latter. W. G. Carey.

Manufacture of [fused] cement. British Portland Cement Manufrs., Ltd., J. G. Baxter, M. K. Bamber, and W. J. Dickinson (E.P. 276,438, 28.5.26).—A rotary kiln adapted for the removal of liquid from the hottest zone is closed at one end, and near this end is provided with holes in its shell, which is tapered from the holes towards the closed end. W. G. Carey.

Accelerating the setting of hydraulic binding agents. Chem. Fabr. Grünau, Landshoff & Meyer A.-G., and W. Kirchner (E.P. 275,897, 5.4.27).—A mixture of calcium chloride or nitrate with aluminium chloride or nitrate is added to the hydraulic binding agent in advance or in the course of working-up the binding agent with other components. Equal parts of the calcium and aluminium salts may be used, or excess of the former, or solutions may be used and the starting and finishing of the setting regulated by varying the concentration or proportions of the solutions.

W. G. Carey.

Preparation of a rapidly-setting cement. Soc. Anon. Cilor (F.P. 611,106, 22.5.25).—In the usual method of grinding together gypsum and blast-furnace slag or other aluminous slag or clinker, the gypsum must be anhydrous. A rapidly-setting cement may be made by grinding the slag and gypsum containing not more than 5% of water separately and subsequently mixing the two powders. A. R. Powell.

Production of a plastic material capable of setting and hardening. R. Schoenhoefer (E.P. 275,788, 5.8.26).—Calcium sulphate is wet-ground until a viscous, very plastic mass is obtained, and is then dried and ground, the processes being repeated as necessary and the amount of water regulated to give a stiff paste for moulding under pressure or a softer paste for casting. Filling and colouring substances are added, and the product may be transformed into burnt gypsum or added to burnt gypsum. W. G. Carey.

Compositions for use as paints, plasters, cements, etc., and treatment of surfaces for the protection, repair, or ornamentation thereof. A. M. Sargint and G. W. Crowe (E.P. 275,772, 9.7.26).—Such a composition is made by incorporating 1 pt. by vol. of zinc oxide or magnesium oxide or a mixture of these and 4 pts. by vol. of a finely-divided metal with a solution of sodium silicate of *d* 1·29, a proportion of basic lead compounds (litharge and white lead) being also added to promote setting or hardening. The composition when *in situ* is treated with a solution of calcium chloride.

W. G. Carey.

Manufacture of bricks, tiles, and other moulded articles made from sand and lime. C. H. Thompson (E.P. 276,597, 11.5.27).—Pressings or mouldings of silicious sand with 6—7·5% of calcium hydrate and colouring matter are treated with lime-kiln waste gases in an autoclave at 3 atm. pressure, and a concurrent or separate treatment with high-pressure steam.

W. G. Carey.

Production of compositions of matter [for lining kilns etc.]. E. C. R. Marks. From G. Polysius Co. (E.P. 276,185, 19.10.26).—A refractory lining for kilns, furnaces, etc. is made by mixing corundum grains (fused bauxite) and aluminous cement in any desired proportions, grinding them together, and baking. The mixture may be introduced directly into the furnace and rammed in to form a lining, or bricks, slabs, etc. made by baking such a mixture. W. G. Carey.

Production of soft pliable wood [by steaming]. Anglo-European Co., Ltd. From Britische Holz-Agentur A.-G. (E.P. 275,058, 25.11.26).

Tar for road-making purposes (F.P. 610,572).—See III.

Slag for use as cement (G.P. 441,365).—See X.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Dissolution of cementite in α-iron and its precipitation. J. H. Whiteley (Iron and Steel Inst., Sept., 1927. Advance copy. 11 pp.).—Numerous tests on specimens of mild steel show that carbon is soluble in α-iron above 630°, the initial temperature for dissolution depending on the purity of the ferrite. The solubility increases with the temperature, and at 720° it is about 0·03%. The carbon can be retained in solid solution on quenching. On tempering, precipitation occurs in the ferrite grains at about 250°. As the temperature rises the minute carbide particles diffuse to the grain boundaries, the rate being very high at 550° and extraordinarily rapid at the A1 point. During slow cooling the dissolved carbide is deposited on existing crystals, and the ferrite in ordinary steel contains very little carbon. Brinell tests showed that the presence of dissolved carbon in α-iron slightly increased the hardness.

T. H. Burnham.

Influence of cold-rolling and subsequent annealing on the hardness of mild steel. C. A. Edwards and K. Kuwada (Iron and Steel Inst., Sept., 1927. Advance copy. 17 pp.)—Specimens of mild steel were subjected to various reductions up to 40% by one or two passes, and the Brinell hardness was determined after annealing for 30 min. at 500—750°. The hardness of cold-working tended to increase as the annealing temperature rose to about 500°. The removal of cold work was most effective between 550—650°, the range being lower the higher the degree of cold work. Specimens subjected to 11·6—19·5% reduction and annealed at 650—750° had a hardness below that of the unrolled material. Hardness-reduction curves were plotted for each annealing temperature. For the unannealed steel there was a break or minimum at about 20% reduction. About 500° the temperature may be only high enough to cause internal strains and the hardness will increase slightly. At a little higher temperature crystal growth took place over a limited range of cold work with a minimum hardness at about 12% reduction by one pass. At 600° a fall in hardness occurred at about the same reduction. After recovery of the hardness curve it fell off again continuously, due to increasing tendency to recrystallisation with increasing cold work. Above 600° crystal growth occurred, due to critical cold work and recrystallisation. After a minimum in the hardness curve it rose again slowly, due to greater recrystallisation and consequent decrease in crystal size.

T. H. Burnham.

Theory of the blast-furnace process. F. Wüst (Iron and Steel Inst., Sept., 1927. Advance copy. 13 pp.).—The gases in the zone in front of the tuyères are of an oxidising nature, and the iron trickling down is oxidised, forming large quantities of ferrous oxide which are taken up by the slag. Reduction of manganous oxide, silica, and phosphoric acid from the slag cannot, therefore, take place, and reduction of these oxides

must occur above the tuyère level. The analyses of washed iron from one coke and five charcoal blast-furnaces are given, and show that it is already a pig iron of the same character as that being produced. Laboratory tests at 900—1200° with electrolytic iron and silica, phosphoric acid, and manganous oxide resulted in the reduction of these oxides in the presence of solid carbon. Hydrogen and carbon monoxide reduced only manganous oxide. In the blast furnace the principal reducing agent is carbon, formed according to the equation $2CO = CO_2 + C$, which penetrates into the pores of the ore. Silicon, manganese, and phosphorus diffuse in the same way into the iron before melting, so that the iron is actually formed into pig iron before it melts. T. H. Burnham.

Constitution and physical properties of alloys of copper, zinc, and cadmium. C. H. M. Jenkins (Inst. Metals, Sept., 1927. Advance copy. 39 pp.).—The effects of small quantities of cadmium on the mechanical properties of 70 : 30 and 60 : 40 brasses have been investigated by means of impact and tensile tests; density determinations were also made. The addition of small amounts of cadmium causes a slight increase in the tensile strengths, but this is counterbalanced by reductions in the elongations; the effect is more marked in the 70 : 30 brass than in the 60 : 40. The presence of 0·13% Cd reduces the workability of the 70 : 30 alloy when hot, but does not affect the 60 : 40 brass, or the 70 : 30 when cold-rolled. If zinc-bearing cadmium is added to molten copper a loss of cadmium occurs, but much is retained. In general, however, zinc containing as much as 0·3% Cd may be used for the preparation of brass without noticeable alteration in the properties. The equilibrium diagram of the copper–zinc–cadmium alloys containing 100—44% Cu and 0—10% Cd has been determined by thermal and microscopical methods. The results are complex, the chief effect being the lowering of the solidus in α-brass to below 600° by comparatively small additions of cadmium (1—2% or over). This gives a wide freezing range and tends to increase unsoundness in these alloys. The solidus of the β and γ constituents of brass is not affected to the same extent. The peritectic reactions α + liquid $\rightarrow \beta$ at 906° in the copper–zinc system, and α + liquid $\rightarrow Cu_2Cd$ at 549° in the copper–cadmium system, are combined in the ternary system to give a reaction β + liquid $\rightarrow \alpha + Cu_2Cd$ at 614°, and this causes marked structural changes affecting the mechanical properties. β-Brass shows a marked increase in its solvent power for cadmium with rise of temperature, but at room temperature if the alloys are slowly cooled very little cadmium remains in solid solution in the α, β, or γ phases. W. Hume-Rothery.

Copper–magnesium alloys. II. W. T. Cook and W. R. D. Jones (Inst. Metals, Sept., 1927. Advance copy. 22 pp. Cf. B., 1926, 830).—The properties of copper–magnesium alloys containing 0—10% Cu and traces of iron and silicon were examined in the forged and heat-treated conditions. The specimens were heated at 400—430° and forged under a steam-hammer; the forging was stopped when the temperature fell to 350°, the specimens being then reheated before the forging was continued. To obtain the best results the forging temperature must be controlled, and must not fall too low, whilst any possibility of cold-shut when casting the ingots for forging must be prevented. The addition of copper to magnesium in the forged condition is beneficial up to about 2% Cu, the hardness, ultimate strength, proof stress, and limit of proportionality all being increased, whilst the ductility is not lowered too seriously. No advantage is gained by the further addition of copper, the increase in tenacity being so small as to be counterbalanced by the increase in density. In general, the elongation and reduction of area of these alloys are low. If the forging temperature is low the ductility is poor, and is not improved by heat treatment, whilst properly forged specimens are not affected to any extent by simple heat treatment.
W. Hume-Rothery.

Equilibrium diagram of copper–tin alloys containing from 10 to 25 atomic per cent. of tin. A. R. Raper (Inst. Metals, Sept., 1927. Advance copy. 15 pp.).—The equilibrium diagram of the system copper–tin has been investigated within the above limits by thermal and microscopical analysis. The boundary between the ($\alpha + \beta$) and β fields has been redetermined, and shows a break at 580° which, with other evidence, confirms the view of Stockdale (A., 1925, ii, 973) that the β phase undergoes a polymorphic transformation denoted $\beta \rightleftharpoons \beta'$. The β' eutectoid point, corresponding to the change $\beta' \rightleftharpoons \alpha + \delta$ is at 16·5 atomic-% Sn and 520°. The γ phase also undergoes a eutectoid transformation, $\gamma \rightleftharpoons \eta + \beta$ at 638° and 23 atomic-% Sn, and the insertion of this new horizontal makes this part of the diagram clear. This eutectoid change takes place very readily, and is not prevented by quenching.
W. Hume-Rothery.

The system magnesium–cadmium. W. Hume-Rothery and S. W. Rowell (Inst. Metals, Sept., 1927. Advance copy. 18 pp.).—The equilibrium diagram of the system magnesium–cadmium has been determined by thermal and microscopical analysis. The system contains a solid solution based on cadmium denoted α, a definite intermetallic compound $MgCd_2$ of fixed composition, and a solid solution in magnesium, denoted β. At ordinary temperatures the limits of the α and β solid solutions are 0—24 and 40—100 atomic-% Mg, respectively. The liquidus and solidus are close together in the whole range of alloys, but do not meet at 50 atomic-% Mg as previously supposed (A., 1906, ii, 355; 1910, ii, 954). The compound $MgCd_2$ is formed from β and liquid by a peritectic reaction at 379°, and itself undergoes a peritectic reaction with the liquid at 358° to form the solid solution α. Prolonged annealing is necessary to secure equilibrium in the region 25—40 atomic-% Mg. The β solid solution undergoes a transformation in the range 40—60 atomic-% Mg at temperatures from 200° to 250°, but no change in microstructure could be detected. The temperature of this change rises to a maximum at 54, and not, as previously reported, at 50 atomic-% Mg.
W. Hume-Rothery.

Under-cooling of some aluminium alloys. (Miss) M. L. V. Gayler (Inst. Metals, Sept., 1927. Advance copy. 28 pp.).—The effect of rapid cooling on the structure of some silicon–aluminium and copper–aluminium alloys.

has been studied. When "normal" aluminium–silicon alloys were cooled at rates of from 1° to 2° per sec., systematic under-cooling was observed, and solubility curves could be constructed which probably approximated closely to the supersolubility curves of aluminium and silicon. The resulting microstructures are discussed in detail. With "modified" aluminium–silicon alloys, however, systematic under-cooling does not occur, and the solubility curve of the "modified" diagram corresponds closely to the supersolubility curves of the normal aluminium–silicon alloys. Under-cooling was also observed in (*a*) aluminium–silicon alloys containing 10% Si and varying amounts of iron, (*b*) copper–aluminium alloys containing 7—38% Cu and traces (0·14%) of iron, and (*c*) copper–aluminium–iron alloys containing 7% Cu and 0·2—0·7% Fe; the changes in structure are described. The "modified" structure could not be obtained by the chill-casting of high-purity aluminium–silicon alloy. W. Hume-Rothery.

Protection of aluminium and its alloys against corrosion. H. Sutton and A. J. Sidery (Inst. Metals, Sept., 1927. Advance copy. 17 pp.).—Samples of aluminium and aluminium alloys were treated by different protective processes, and their resistance to corrosion by sea water was determined by "wet and dry" and "spray" tests. The anodic oxidation process gave considerable protection to both aluminium and its alloys, especially when the process was followed by the application of grease (lanoline). The increase of weight caused by this process is negligible compared with that involved by plating, but parts in contact with other metals cannot be treated anodically. The "throwing power" of the anodic process is good, and parts of irregular shape can be treated without special arrangements. The limiting radius of bending of thin sheets is slightly decreased by the brittle anodic film, the insulating properties of which cause difficulty in securing electrical contact. Nickel plating is unsatisfactory, but zinc and cadmium deposits, 0·0005 in. thick, give good protection, with the single exception of cadmium on aluminium. Deposits of this thickness weigh about 0·6—0·74 oz./ft.^{2}, and the protection is due to a film of mixed oxides, hydroxides, or basic salts. The samples examined were aluminium, duralumin, B.E.S.A. alloys L5 and L24, a modified aluminium–silicon alloy, and an aluminium–zinc alloy containing 14·86% Zn. W. Hume-Rothery.

Nature of the film produced by anodic oxidation of aluminium. H. Sutton and J. W. W. Willstrop (Inst. Metals, Sept., 1927. Advance copy. 5 pp.).—Samples of anodically treated aluminium were heated in dry hydrogen chloride at 300—320°; the metallic aluminium volatilised and the surface film was left unchanged. The films isolated from commercial aluminium contained small quantities of elementary silicon and were usually grey, owing to traces of carbon. The films obtained were from 0·74 μ to 2·10 μ thick, being from 2 to 6 times as thick as those produced by heating aluminium at 600° in oxygen for 80 hrs. (cf. Pilling and Bebworth, B., 1923, 359 A), and from 100 to 300 times as thick as the atmospheric corrosion films of Vernon (2nd Rept. Atmospheric Corrosion Research Committee). From the volume of gas evolved by heating *in vacuo* at 1200°, the film is shown to consist of oxide and not hydroxide. W. Hume-Rothery.

Determination of lithium in scleron and similar aluminium alloys. E. Schürmann and W. Böhm (Chem.-Ztg., 1927, **51**, 677—678, 698—699).—The alloy (20 g.) is dissolved in dilute hydrochloric acid with the addition of a little hydrogen peroxide when action has ceased. The solution is diluted to 400 c.c., cooled, and saturated with hydrogen chloride, and the precipitate of $AlCl_3,6H_2O$ is collected on a porous glass filter, washed with hydrochloric acid (*d* 1·19), dissolved in water, and reprecipitated as before. The combined filtrates are evaporated to 100 c.c., cooled, and again saturated with hydrogen chloride. The filtrate from the second precipitate of aluminium chloride is evaporated to dryness on the water-bath, and the residue repeatedly moistened and dried to remove free acid. Two procedures are now available. (A) The solution of the chlorides obtained as above is treated at 60—70° with 5 g. of oxalic acid dissolved in a little water, the zinc and copper oxalates are collected, washed, redissolved by heating with nitric and sulphuric acids until a dry residue is obtained, and reprecipitated with oxalic acid, the combined filtrates being evaporated with nitric and sulphuric acids to complete dryness. The residue is dissolved in water, the remainder of the zinc precipitated with hydrogen sulphide in formic acid solution, the iron separated as basic acetate and the manganese as dioxide by electrolysis, and the filtrate treated with ammonia and hydrogen peroxide to remove the last traces of alumina and manganese. The solution is then evaporated to dryness and the residue heated to remove ammonium salts. The sulphates of lithium and sodium together with a little magnesium are thus obtained; they are converted into acetates by treatment with lead acetate, the lead sulphate is collected on a filter, and the filtrate treated with hydrogen sulphide. The filtered solution is evaporated nearly to dryness with hydrochloric acid and the lithium chloride extracted with amyl alcohol. The alcoholic solution is evaporated to dryness with a little sulphuric acid, the residue heated at dull redness, and weighed as lithium sulphate. (B) Zinc, copper, iron, manganese, and magnesium are separated from the neutral solution obtained after removing the aluminium by digesting the liquid at 70° with the silver carbonate obtained from 20 g. of silver nitrate by precipitation with sodium carbonate. The precipitate is collected (the filtrate should be free from chloride), washed with hot water, and discarded. The filtrate is evaporated with hydrochloric acid and extracted with amyl alcohol, and the analysis finished as in (A). A. R. Powell.

Patents.

[Pit] furnace [for ingots]. G. Kehren (E.P. 273,671, 27.5.27. Conv., 30.6.26).—A pit furnace for reheating ingots by means of powdered fuel is provided with a separate combustion chamber, but the two chambers are parallel to each other with no dividing wall, so that the ingots are exposed to direct radiant heat from the combustion chamber. B. M. Venables.

Recovery of iron oxide from blast-furnace flue dust. M. A. Minot (F.P. 612,394, 27.6.25).—An iron oxide pigment may be made from blast-furnace flue dust

by mixing it with 50% by wt. of sulphuric acid (*d* 1·58), heating the mixture at 450—600° until fumes cease to be evolved, and elutriating the product.

A. R. Powell.

Production of pig iron and a slag suitable for direct use as a cement. Vereinigte Stahlwerke A.-G. (G.P. 441,365, 22.1.24).—Low-grade iron ores are smelted in a blast furnace with fluxes to yield a highly calcareous or aluminous slag and pig iron. To obtain the necessary temperature the blast is enriched with oxygen. A. R. Powell.

Production of synthetic pig iron. M. Frankl (E.P. 252,708, 19.5.26. Conv., 29.5.25).—For the production of pig iron from steel scrap and turnings the turnings are melted with suitable quantities of fuel and sand in an acid furnace so that carbon dioxide first formed is quickly reduced to carbon monoxide. The scrap is introduced into a basic furnace heated by combustion of the excess carbon monoxide from the acid furnace, and molten metal is mixed with and carburised by the metal at the base of the acid furnace, after which the mixed metal is desulphurised in a separate container by slag from the basic furnace. C. A. King.

Manufacture of grey cast iron. H. Hanemann (E.P. 262,043, 18.5.26. Conv., 25.11.25).—Grey iron containing flaky graphite is produced by maintaining the molten iron in a liquid condition, *e.g.*, for 1 hr. at 1250—1300°, until all the graphite grains are in solution, and then casting the metal in the usual manner.

C. A. King.

Re-melting chromium-steel scrap. W. M. Farnsworth, Assr. to Central Alloy Steel Corp. (U.S.P. 1,641,326, 6.9.27. Appl., 24.8.26).—When remelting high-chromium, low-carbon steel scrap, ferrosilicon is added with the charge to maintain a strongly reducing condition and to prevent absorption of carbon by the bath. C. A. King.

Manganese steel. (Sir) R. A. Hadfield (E.P. 276,048, 11.5.26).—To mitigate the disadvantages of producing steels of higher phosphorus content for special track structures for railways, manganese steels containing 11% (or more) Mn, 1·6% C, and less than 0·065% (0·02—0·03%) P are claimed, and a method of their manufacture is described. C. A. King.

Alloy steel. Cammell, Laird & Co., J. McN. Allan, and A. P. Hague (E.P. 276,249, 26.2.27).—Alloy steel possessing strength and resistance to scaling at high temperatures and not liable to warp or crack on cooling contains 46·45—57·05% Fe, 0·35—0·55% C, 0·3—0·5% Si, 0·3—0·5% Mn, 26—30% Ni, and 16—22% Cr. The alloy is suitable for case-hardening retorts, annealing boxes, furnace grates, and fire bars.

C. A. King.

Casting high-melting-point metals. R. B. Dale (E.P. 276,210, 9.12.26).—The cooling of molten iron or steel in moulds is controlled by heat exchange with a liquid of high b.p., *e.g.*, mercury or diphenyl oxide, at the same time allowing a high head of molten metal to exert a pressure on the cooling ingot. In cases in which chilling is relatively unimportant, the fluid may be sprayed on to the mould. Rotary moulds are rotated at such a speed that the centrifugal force is equivalent to the pressure supplied by the head of molten metal.

C. A. King.

Steel-hardening bath. O. Kröning and R. Boës (E.P. 276,289, 30.5.27).—A hardening bath for steel consists of a mixture of 94% of juice of onions or similar alliaceous plants, 4% of acetic acid, and 2% of a solution of calcium chloride. C. A. King.

Hardening bath for machine tools. Glockenstahlwerke A.-G. vorm. R. Lindenberg, Assees. of W. Schröder (G.P. 441,530, 6.12.25).—Iron and steel machine tools are hardened by heating them to the desired temperature in a bath of molten aluminium or one of its alloys. A. R. Powell.

Oxidation-resisting material [iron-aluminium-titanium alloy]. R. F. Flintermann, Assr. to Gen. Electric Co. (U.S.P. 1,641,752, 6.9.27. Appl., 10.10.19).—A ferrous alloy contains approximately 19·5% Al and 1—5% Ti. F. G. Crosse.

Method of coating metal by dipping. E. C. R. Marks. From American Machine & Foundry Co. (E.P. 276,208, 3.12.26).—In the process of coating metals with lead only sufficient tin, about 3%, is introduced into the bath to increase the solubility of phosphorus in lead. As the process proceeds phosphorus is lost from the bath, and the necessary quantity is maintained by the introduction of phosphor-lead from time to time.

C. A. King.

Method of treating lead dross. O. P. Chisholm, Assr. to Amer. Smelting & Refining Co. (U.S.P. 1,642,358, 13.9.27. Appl., 17.4.26).—Lead dross obtained from the refining kettle is smelted with fluxes and scrap iron or lead blast-furnace speiss to obtain metallic lead, iron speiss, copper matte, and a slag.

A. R. Powell.

Method of treating [sulphide] ores. F. M. Simonds, Assr. to A. F. Hyde (U.S.P. 1,637,838, 2.8.27. Appl., 27.1.25).—Ground sulphide ores are heated at a relatively low temperature in a reducing or neutral atmosphere and then in a slightly oxidising atmosphere partially to burn the sulphur, causing an increase in temperature and volatilisation of sulphur.

C. A. King.

Production of mercury. W. Glaeser (U.S.P. 1,637,481, 2.8.27. Appl., 14.11.24).—Finely-divided cinnabar ore is treated with dilute hydrochloric acid and an alkali or alkaline-earth hypochlorite at 70°, the solution thus obtained being treated with iron or other suitable metal to form an amalgam, from which mercury is distilled. T. S. Wheeler.

Removing grease from metal parts. Soc. Anon. des Établ. A. Lendormy (F.P. 616,407, 20.5.26).—The greasy parts are cleaned with a soap obtained by the saponification of fats with a high content of hydroxycarboxylic acids. A. R. Powell.

Electrolytic process [for obtaining pure nickel]. Internat. Nickel Co., Assees. of R. L. Suhl, J. W. Sands, and O. B. J. Fraser (Can. P. 264,172, 21.11.25).—Nickel free from cobalt is obtained by the electrolysis of cobaltiferous nickel in a suitable bath of nickel salt, provided that the electrolyte is continuously circulated

from the cell through a purifying vessel, where it is treated with hydrated nickel peroxide to remove cobalt, then back to the cell. A. R. POWELL.

Bearing. Alloy structures. H. M. WILLIAMS and A. L. BOEGEHOLD, Assrs. to GEN. MOTORS RESEARCH CORP. (U.S.P. 1,642,347—9, 13.9.27. Appl., [A] 17.8.22, [B] 19.1.23, [C] 18.4.23).—See E.P. 230,896, 210,063, and 214,580; B., 1925, 509, 288.

Separating the components of alloys. C. G. BOSSIÈRE and H. ZANICOLI (U.S.P. 1,642,574, 13.9.27. Appl., 15.12.24. Conv., 23.10.24).—See E.P. 241,880; B., 1926, 590.

Flotation separation (E.P. 275,778).—See I.

Kiln for calcining ores (E.P. 276,066).—See IX.

XI.—ELECTROTECHNICS.

Electrical resistance of wood as a measure of its moisture content. A. J. STAMM (Ind. Eng. Chem., 1927, 19, 1021—1025).—The relation of the electrical resistance of wood drier than the fibre saturation point was investigated using lead-faced brass clamps as electrodes. Clamping had to be as firm as could be effected by hand to eliminate resistance at the contact. It was found that variation of species of wood, density and size of specimen, or ash content was without importance. On the other hand, a surface film of moisture or a steep moisture gradient in the sample introduced large errors. The resistance increased 1·8 times for each 1% reduction in moisture content. The practical application of the method is evidently limited to thoroughly seasoned wood which has been kept for some time under fairly constant humidity conditions. C. IRWIN.

Ignition of gases by hot wires. SHEPHERD and WHEELER.—See II.

Magnetic changes in steels. DICKIE.—See X.

Cathodic disintegration. SMITH.—See X.

PATENTS.

Primary battery. W. A. F. BLEECK (E.P. 248,011, 23.2.26. Conv., 23.2.25).—Sodium silicate is added to the electrolyte of a battery of the double-fluid type described in E.P. 5297 of 1908 and 15,194 of 1909 (cf. B., 1908, 1025; 1909, 1144). J. S. G. THOMAS.

Electrolyte for lead accumulators. Galvanic cell. R. E. BRENNER (F.P. 613,509—10, 23.7.25). —(A) Dilute sulphuric acid is mixed with the sulphate of a metal which forms a number of oxides, *e.g.*, chromic sulphate, an ester of sulphuric acid, *e.g.*, ethyl sulphate, and a small proportion of a so-called catalyst, *e.g.*, uranium oxide, UO_2. Sulphation of the electrodes is reduced by use of such electrolyte. (B) The galvanic cell comprises a carbon anode immersed in a nitrate solution containing hydrochloric acid and/or sulphuric acid, a cathode of aluminium or aluminium alloy immersed in an alkaline fluid containing, if necessary, an alkali chloride, and a semi-permeable diaphragm, *e.g.*, containing cupric chromate. The voltage of the cell is 2·5—2·7; it can be used at temperatures between 40° and 100°. J. S. G. THOMAS.

Manufacture of lead electrode for accumulators. A. STRASSER (U.S.P. 1,641,241, 6.9.27. Appl., 15.8.25. Conv., 23.8.24).—An alloy of lead with an alkali metal capable of reducing water, placed in a hollow perforated insulating container, is treated with caustic soda lye, then with water to extract the alkali metal, and finally is subjected to the action of an electric current in an electrolyte. J. S. G. THOMAS.

Electrode for rectifiers and electrolytic condensers. E. F. ANDREWS, Assr. to ANDREWS-HAMMOND CORP. (U.S.P. 1,637,795, 2.8.27. Appl., 10.9.26).—A film-forming electrode is composed of equal parts of magnesium and aluminium with addition of sufficient cadmium to give suitable mechanical properties. It is used in an electrolyte of potassium hydroxide in conjunction with a nickel electrode. T. S. WHEELER.

Electroresponsive cell. C. H. ZIERDT, Assr. to UNION SWITCH & SIGNAL CO. (U.S.P. 1,641,564, 6.9.27. Appl., 14.12.22. Renewed 22.1.25).—A thermo-sensitive cell, the resistance of which increases considerably with increase of current, and which can be used as an automatic cut-out in a series lighting system is made of powdered cuprous oxide and sodium silicate, moulded together and baked to a solid mass. J. S. G. THOMAS.

Production of chemically pure coatings of carbon on the electrodes of vacuum discharge vessels, more especially mercury vapour rectifiers. W. DÄLLENBACH (G.P. 441,378, 21.11.24).—Gaseous material, *e.g.*, acetylene, used for the production of the coating is discharged into the vacuum vessel through one or more channels in the immediate neighbourhood of the active electrode surfaces, and is chemically decomposed by the electric discharge. J. S. G. THOMAS.

Electric arc for treating gases. AKTIESELSKAPET NORSK STAAL (ELEKTRISK-GAS-REDUKTION) (G.P. 441,223, 21.10.25).—The electrode on the gas outlet side consists of a bath of fused material, such as heat-resisting oxides and/or salts, or a metal covered with a relatively thin layer of oxides or salts. L. A. COLES.

Thermocouple. O. HERMANN, Assr. to THERMO ELECTRIC BATTERY CO. (U.S.P. 1,637,822, 2.8.27. Appl., 9.9.26).—The positive element is formed of nickel, cobalt, and copper, and the negative of nickel, cobalt, and molybdenum. T. S. WHEELER.

Metallising process [for textiles etc.]. Metallising furs and feathers. J. VON BOSSE, K. RICHTER, K. LAUCH, H. SIEGELBERG, and W. KOCH (E.P. 252,710 and 276,291, [A, B] 20.5.26. Conv., [A] 27.5.25).—The textiles, furs, etc. are exposed *in vacuo* to cathode radiation from cooled electrodes, *e.g.*, of gilded copper. J. S. G. THOMAS.

Method of [nickel] plating. H. GARDNER, Assr. to FORD MOTOR CO. (U.S.P. 1,642,238, 13.9.27. Appl., 19.2.23).—A solution for a nickel-plating bath is made by adding to 1 gal. of water 8 oz. of nickel ammonium sulphate, 4 oz. of nickel sulphate, 2 oz. of boric acid, $\frac{1}{16}$ oz. of ammonium chloride, 0·15 oz. of cadmium chloride, and 1·5 oz. of glycerin. F. G. CROSSE.

Electric furnace. D. CROESE (U.S.P. 1,642,359, 13.9.27. Appl., 1.12.26. Conv., 27.8.25).—See E.P. 266,658; B., 1927, 302.

Splitting fluid hydrocarbons (E.P. 275,281).—See II.

Insulating composition (E.P. 275,795).—See V.

Nitric acid (G.P. 440,657). **Cuprous oxide** (G.P. 440,755).—See VII.

Electrolytic process (Can. P. 264,172).—See X.

Varnishes (E.P. 275,813).—See XIII.

Treatment of seeds (E.P. 268,291).—See XVI.

XII.—FATS; OILS; WAXES.

[Soap] cakes obtained by the neutralisation of oils. VIZERN and GUILLOT (Ann. Chim. analyt., 1927, [ii], **9**, 257—261).—The technical process consists in neutralising the oil with sodium hydroxide solution at 40—50°, raising the temperature to 70°, and salting out. The cake obtained, which is invariably acid, is a mixture of hydrated soap, neutral oil, and free fatty acid. The oil which separates from the residual liquid after breaking down the emulsion is always less acid than that retained by the soap. To ascertain the composition of the cake, determinations are made of the total fatty material, the fatty acids combined as soap (S) by titration of the alkalinity of the ash, the acidity of free fatty acids, and the acidity of the total fatty acids (A), the last three being calculated per 100 g. of total fatty material. Neglecting the small amount of glycerin formed, the neutral oil in the cake is given by ($100—A$), and the free fatty acids by ($A—S$). F. R. ENNOS.

Glycerol content and specific gravity of technical glycerin solutions. W. PRAGER (Chem.-Ztg., 1927, **51**, 589—590).—Stiepel's formula for calculating the glycerol content of solutions from the sp. gr. and ash content of the latter may lead to erroneous results through taking no account of the density of the ash or of the content and sp. gr. of any organic matter present. Terms allowing for these factors can easily be introduced into the equation connecting the content of glycerol with the sp. gr. of the solution. W. T. K. BRAUNHOLTZ.

Determination of the constitution of tung oil fatty acids by spectroscopic methods. W. MANECKE and F. VOLBERT (Farben-Ztg., 1927, **32**, 2829—2831, 2887—2889).—A general outline of the measurement of absorption spectra is given. The absorption curves obtained for elæostearic acid and its methyl ester (characterised by a deep band at λ2650—2750 Å.) differ markedly from the curves for linoleic acid and its esters, which resemble those for oleic and crotonic acids, with slight increase in the intensity of absorption and displacement of the characteristic weak absorption band in the direction of increasing wave-length. From comparison with saturated fatty acids, this is shown to be the general tendency with increase in degree of unsaturation. Optically pure linolenic acid for comparison is not available, but elæostearic acid is definitely not isomeric with linoleic acid, and must contain more than two double linkings, questions of conjugation and position being unsolved at present. S. S. WOOLF.

Decomposition of vegetable waxes. A. MAILHE (Bull. Soc. chim., 1927, [iv], **41**, 1056—1061).—Japanese wax (from trees of the genus *Anacardiaceæ*) and wax from the Brazilian carnauba tree were dry distilled in presence of zinc chloride (cf. B., 1924, 719). The latter wax evolved a gas rich in hydrogen and methane, and produced an artificial petroleum containing paraffins and olefines, from which colourless light oils, kerosene, heavy oils, and paraffin were isolated. Japanese wax yielded a gas rich in methane and hydrogen, and also containing a large proportion of carbon monoxide, together with an oil containing hydrocarbons having chains of eight to eighteen carbon atoms. Apparently the glycerides in the wax are saponified into acraldehyde and higher fatty acids; the latter decompose into heavy hydrocarbons which, on cracking, produce the oil. The oil contains a fraction of b.p. 60—150°, light oil of b.p. 150—290°, heavy oils, and paraffin. S. K. TWEEDY.

PATENTS.

Treating crude oils and fats. METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 275,536, 20.9.26. Conv., 9.8.26).—Crude oils or fats obtained by pressing, extraction, melting out, etc. are subjected to intensive dehydration *in vacuo* at comparatively low temperatures before filtration and storage. Filtration is rendered easier, and any matter retained by the filtered oil remains in suspension. S. S. WOOLF.

Gas from heavy oils (E.P. 255,423 and 271,899).—See II.

Removing grease from metal parts (F.P. 616,407). See X.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Paints for compass bowls and discs. H. L. SMITH (J. Sci. Instr., 1927, **4**, 314—317).—Paints applied to compass bowls tend to become discoloured, to flake and peel, or to cause discoloration of the alcohol used to fill the bowls. This is attributed to solvent action of the alcohol on the paint, and to the presence of sulphur extracted by the alcohol from the vulcanised rubber rings sealing the bowls. Oil paints and nitrocellulose paints are in general prone to these drawbacks, although satisfactory results were obtained with the following two paints:—(*a*) 50 g. of white lead, 20 g. of "litho middle varnish," 20 c.c. of white (petroleum) spirit, and 5 g. of lead tungate; (*b*) 150 g. of antimony white (a stable variety), 10 g. of pyroxylin, and 180 c.c. of amyl acetate. Water paints, particularly of the casein-gum arabic type, are suitable for this work when freshly prepared, but cannot be kept in the mixed condition. White lead may not be used in the pigment if the vehicle contains much casein, owing to the sulphur content of the latter. S. S. WOOLF.

Principle for testing the durability of paints as protective coatings for wood. F. L. BROWNE (Ind. Eng. Chem., 1927, **19**, 982—985).—The weathering of wood is principally due to internal stresses set up by fluctuating moisture content. Paint protects wood by retarding the rate of passage of moisture in or out. This quality was measured by subjecting painted panels to the following cycle: (*a*) 2 weeks at 60% humidity, (*b*) 2 weeks at 95—100% humidity, (*c*) 2 weeks at 60% humidity, (*d*) 2 weeks' exposure to weathering influences, and so repeatedly. The absorption of moisture in

stage (*a*) gave a measure of the effect of weathering in stage (*c*). It was found that for the first few months the resistance of all paints improved; afterwards, sooner or later, deterioration set in. Until the wood is appreciably exposed the moisture absorbed is independent of the kind of wood. This method is recommended for the comparison of the protective qualities of paints in preference to observations of the time when disintegration becomes visible, which are misleading. C. Irwin.

Uniform varnish films for exposure tests. H. A. Gardner and G. G. Sward (Ind. Eng. Chem., 1927, **19**, 972—974).—It was desired to ascertain whether varnish films of uniform thickness could be made with certainty by spinning on metal panels. The relation of thickness of film to speed of spinning, duration of spinning, viscosity of the varnish, and proportion of non-volatiles in the varnish was therefore determined. None of these relations is linear. The formula $F = 0 \cdot 4N + V^4 + 3$ is suggested, where F is thickness in microns, N is percentage of non-volatiles, and V is absolute viscosity in poises. This is approximately true for 60 sec. spinning at 290 r.p.m. For other times and speeds factors must be introduced. C. Irwin.

Effect of thinners on the consistency of nitrocellulose solutions. P. E. Marling and J. M. Purdy (Ind. Eng. Chem., 1927, **19**, 980—981).—The consistency of nitrocellulose solution diluted with various proportions of turpentine, toluene, xylene, and butyl acetate was measured with a mobilometer. ½-, 4-, and 10-sec. nitrocellulose solutions all suffered a reduction of consistency which was greatest with butyl acetate (a solvent) and least with turpentine. The effects of toluene and xylene were closely similar, and varied according to the concentration of nitrocellulose. C. Irwin.

Solvent balance [in nitrocellulose lacquers]. B. K. Brown and C. Bogin (Ind. Eng. Chem., 1927, **19**, 968—971).—The classification of nitrocellulose solvents and diluents according to their b.p. is misleading, as this bears no definite relation to their volatility at room temperature. The evaporation rate, which is what is really wanted, whilst it varies with the vapour pressure at room temperature and the mol. wt., is best determined empirically. The dilution ratio, or volumes of diluent which can be added to 1 vol. of solution without causing coagulation, is also affected by numerous variables and is wholly empirical. As it varies with concentration, determinations to be useful should be from mixtures of final concentration similar to true lacquers (8—12% of nitrocellulose). A number of such dilution ratios are given. The presence of other liquids in the lacquer also affects the dilution ratio, as is shown by examples. Both factors require consideration, as the dilution ratio must not be exceeded at any time during drying. C. Irwin.

Dilution ratios of nitrocellulose solvents. J. G. Davidson and E. W. Reid (Ind. Eng. Chem., 1927, **19**, 977—980; cf. B., 1926, 714).—The dilution ratios of a number of classes of compounds with respect to a 1 in 3 solution of dry nitrocellulose at 20° were determined. It was found that the glycol ethers have higher dilution ratios to toluene than the butyl esters, but less difference exists in the invariably lower dilution ratio to light petroleum. In this case variation occurs with the distillation range of the petroleum, and the figure is higher for cracked than for straight-run petroleum. A range of 80—120° is recommended, as higher-boiling fractions result in an unbalanced mixture during drying. The dilution ratio decreases as the concentration of nitrocellulose increases. C. Irwin.

Resins in coal. Cockram and Wheeler.—See II.

Patents.

Titanium pigment. P. A. Zuber and M. Billy (F.P. 613,492, 22.7.25).—Finely-powdered titanium ore is mixed with sodium carbonate equivalent to its titanium content, and heated until the mass sinters, fusion being avoided. The product is powdered and heated at 150° with slightly diluted sulphuric acid, which dissolves the titanium, leaving a residue of iron oxide. The solution is hydrolysed for recovery of titania, which is intimately mixed with the requisite amount of barium or calcium carbonate to yield the corresponding titanate, and the mixture is heated at 1100°. A. R. Powell.

Manufacture of drying varnishes. H. Becker, and Siemens & Halske A.-G. (E.P. 275,813, 29.9.26).—Hydrogenated naphthalenes in the form of vapour are subjected to the action of the electric discharge, 1% of a readily frothing substance being present. The charged product, either dissolved in excess of raw material (as obtained), or separated from the latter and redissolved in light petroleum, is used as a varnish. S. S. Woolf.

Pyroxylin lacquer composition. B. K. Brown and C. Bogin, Assrs. to Commercial Solvents Corp. (U.S.P. 1,641,529, 6.9.27. Appl., 12.12.24).—The use of butyl stearate as a pyroxylin lacquer constituent is claimed. S. S. Woolf.

Manufacture of high-grade rosins. Hercules Powder Co. (E.P. 275,862, 22.1.27. Addn. to E.P. 253,082; B., 1926, 988).—As selective solvent for the colouring matter in low-grade rosin, any one of the following may be used in place of furfuraldehyde (cf. *loc. cit.*): a phenol, a chlorohydrin, furfuryl alcohol, sulphur dioxide, aniline. S. S. Woolf.

Production of aldehyde-amine condensation products. L. Light (E.P. 275,725, 12.5.26).—Equimolecular proportions of formaldehyde and a primary aromatic amine are condensed in the presence of specified amounts of acid condensing agents for 5 min. at 70°, after which the temperature is raised to 90° for several minutes. The resinous residue after removal of supernatant aqueous liquor is converted by further heat treatment into hard, transparent, pale yellow to orange resinous products, which in turn may be converted into brown opaque resins by treatment with boiling water. S. S. Woolf.

Manufacture of resinous condensation products from aryloxyacetic acids and aldehydes. I. G. Farbenind. A.-G. (G.P. 439,962, 26.3.20. Addn. to G.P. 364,040; B., 1923, 613 a).—Partly condensed aldehyde-phenol products are heated with alkali and chloroacetic acid, β-bromopropionic acid, or other halogenated aliphatic acids. Fusible carboxylated resins,

soluble in the weakest alkalis (*e.g.*, borax), are obtained which retain this solubility after hardening. C. HOLLINS.

Manufacture of resinous condensation products from dihydroxydiphenylethane and reactive methylene compounds. L. H. BAEKELAND, Assr. to BAKELITE CORP. (U.S.P. 1,637,512, 2.8.27. Appl., 30.6.22).—Dihydroxydiphenylethane can be condensed at about 180° with relatively small amounts of hexamethylenetetramine or paraformaldehyde, to give resins of the phenol-formaldehyde type. T. S. WHEELER.

Manufacture of polymerised vinyl esters. CONSORTIUM FÜR ELEKTROCHEM. IND. G.M.B.H. (E.P. 261,406, 13.11.26. Conv., 14.11.25. Cf. B., 1926, 680).—When polymerisation of a vinyl ester has been initiated by heat in a vessel fitted with a reflux condenser, a further batch of unpolymerised substance is run in, while a part of the reaction mixture is run into a second similar apparatus, the temperature and energy of the reaction being thereby controlled. The reaction may be effected in the presence of liquids, diluent or otherwise, *e.g.*, alcohol, acetic acid, acetal, etc.; or in the presence of gases, inert (nitrogen) or active (oxygen). Examples are given of the polymerisation of vinyl acetate containing 1% by wt. of benzoyl peroxide and of a mixture of vinyl acetate with glacial acetic acid, acetaldehyde, paraldehyde, and potassium acetate. B. FULLMAN.

Production of moulded articles and plastic materials. H. BURMEISTER (E.P. 275,678, 10.2.26).—Dry mixtures of resorcinol or other polyhydric phenols, solid polymerides of formaldehyde, and, if desired, filling materials are converted into moulded articles by a single hot-pressing process. The reagents may be first melted and mixed with the fillers as an intermediate stage. S. S. WOOLF.

Colloidally dispersed material (E.P. 246,874).—See I.

Iron oxide from flue dust (F.P. 612,394).—See X.

Protecting photographic films etc. (U.S.P. 1,640,148).—See XXI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

New chemical reactions of rubber hydrocarbons. Reactions with metallic halides. H. A. BRUSON, L. B. SEBRELL, and W. C. CALVERT (Ind. Eng. Chem., 1927, **19**, 1033—1037).—Solutions of rubber in benzene when treated with stannic chloride in an atmosphere of nitrogen afford a reddish-brown additive *compound*, $(C_5H_8)_{10}$,$SnCl_4$, slowly decomposed in moist air, which when heated at 170° suddenly swells and decomposes with evolution of stannic chloride. On decomposition under nitrogen with 95% alcohol, white amorphous polymerides of rubber are obtained which are less unsaturated and have a higher m.p. or decomposition point than rubber itself. It is suggested that addition takes place on the secondary valencies of the stannic chloride in such manner that upon treatment with alcohol some of the double linkings of the hydrocarbon bridge across to form rings in the more reactive positions of the double molecule, leaving other unsaturated linkings intact. It is in accordance with this theory that in carbon tetrachloride or chloroform solution, titanium tetrachloride, possessing two secondary valencies, gives a coloured additive product, affording, like the stannic chloride compound, both soluble and insoluble polymerides, whereas ferric chloride, with three secondary valencies, and antimony pentachloride, with one, afford additive compounds which yield only insoluble polymerides. High-grade, unmilled, plantation *Hevea* pale crêpe affords a mixture of two isomeric additive compounds $(C_5H_8)_{10}$,$SnCl_4$, which on decomposition yield two isomeric hydrocarbons, one (75% yield) readily soluble in cold benzene, and the other (25% yield) insoluble in all the usual solvents. If, however, the original rubber is first subjected to an ether-diffusion process (cf. Feuchter, B., 1925, 557), only the soluble polymeride is obtained; attempts to prepare the insoluble polymeride from the ether-insoluble phase gave unsatisfactory results owing to the concentration of ash and protein in this fraction. *Hevea* rubber is therefore shown to consist of two distinct isomerides, thus confirming physical evidence already obtained. Cold overmilled rubber afforded the same soluble and insoluble polymerides in unchanged relative properties; rubber milled at 100° for 45 min. gave both polymerides, whereas rubber which had been heated in an inert atmosphere for 24 hrs. at 163° in the solid state or in solution at 80° afforded only the soluble polymeride. In the case of "dead-milled" rubber the insoluble polymeride could only be made to separate by cooling to 5°. The soluble polymeride softens at 220—225°, m.p. about 280° (decomp.). Its solutions up to 50% strength are non-viscous. Exact values for unsaturation could not be obtained. The insoluble polymeride sinters at about 255° and decomposes above 300°. Isoprene reacts similarly with stannic chloride affording a soluble polymeride which softens at 110°, m.p. above 135° (decomp.). Polymerised isoprene yields a polymeride softening at 175—180°, and balata a product, $(C_5H_8)_x$, which softens at 200°, m.p. 250° (decomp.). (Cf. A., 1926, 719; B., 1926, 451.) R. BRIGHTMAN.

Accelerator characteristics as revealed by coefficients of vulcanisation. A. F. HARDMAN and F. L. WHITE (Ind. Eng. Chem., 1927, 19, 1037—1040).—The relation between time of cure and combined sulphur (coefficient of vulcanisation) and also tensile at break was determined for twelve accelerators. For one class, *e.g.*, triphenylguanidine, the sulphur curve is a straight line though the tensile curve shows flattening. For a second, *e.g.*, zinc ethylxanthate, the sulphur curve becomes horizontal in 30 min. A third class—the remaining accelerators—is intermediate. The transient accelerators such as zinc ethylxanthate are believed to decompose quickly by the influence of heat or by reaction with sulphur etc. C. IRWIN.

Perchloric acid as an oxidising agent for the determination of sulphur in rubber. E. KAHANE (Ann. Chim. analyt., 1927, [ii], 9, 261—264).—Perchloric acid mixed with nitric acid forms an effective agent for the oxidation of rubber; the reaction will not be excessively violent provided small quantities of rubber (not more than 2 g.) are used. The organic matter is oxidised in two stages, the soluble nitro-derivatives first formed being subsequently oxidised by the perchloric acid. The presence of perchloric acid does not affect the accuracy

of the determination of sulphur as barium sulphate. For the determination, 1 g. of rubber is treated with 10 c.c. of nitric acid (*d* 1·33) and 5 c.c. of perchloric acid (*d* 1·61) in a 250 c.c. Kjeldahl flask, and the whole gently heated until the rubber dissolves, and then boiled to expel nitrous fumes. Boiling is continued until the dark solution becomes colourless and the larger part of the perchloric acid is removed, the whole operation usually occupying 7—8 min. The solution is then diluted, filtered, and the sulphate determined by means of barium chloride in the usual manner. F. R. Ennos.

Patents.

Vulcanisation of rubber. Chem. Fabr. Kalk G.m.b.H., and H. Oehme (E.P. 272,860, 21.3.27. Conv., 21.6.26).—Zinc oxide produced from the precipitated hydrated oxide or from easily decomposed inorganic or organic zinc salts by heating at a low temperature has little or no covering power in linseed oil, but is much superior to pyrogenic zinc oxide in its beneficial influence on the vulcanisation of rubber and on the tenacity of the product. D. F. Twiss.

Manufacture of vulcanisation accelerators and products obtained thereby. E. C. R. Marks. From E. I. Du Pont de Nemours & Co. (E.P. 276,435, 28.5.26).—An aldehyde, a primary amine, and carbon disulphide are made to react in any order, definite compounds being obtained when the mol. ratios are 2 : 2 : 1, 2 : 1 : 1, 4 : 2 : 1, or 1 : 1 : 1. The products are vulcanisation accelerators. From equimolecular proportions of the reagents, dimethylene-1 : 3-thiaimines of the type, $R{\cdot}CH\langle{}^{NR'}_{S}\rangle CS$, are obtained; from 2 mols. of aldehyde, 2 mols. of amine, and 1 mol. of carbon disulphide "carbothialdines" (cf. Mulder, A., 1874, 47; Delepine, A., 1897, i, 457) result, to which the structure, $R{\cdot}CH:NR'\langle{}^{CHR\cdot NR'}_{S\text{———}CS}$, is assigned. The following combinations, with 1 mol. of carbon disulphide, are described: butaldehyde and methylamine (2 : 2); acetaldehyde and aniline (2 : 2); butaldehyde and *n*-butylamine (2 : 2); butaldehyde and aniline (2 : 1, and 4 : 2); butaldehyde and methylamine (1 : 1). C. Hollins.

Method of improving the properties of gutta-percha, balata, etc. Felten & Guilleaume Carlswerk A.-G. (G.P. 440,122, 27.6.25).—The natural resins occurring in the crude latex are wholly or partly removed by known methods, hardened by treatment with lime or other metallic oxide or hydroxide, and re-mixed with the remainder of the latex. The natural resins may also be replaced by hardened resins from other sources. The products so obtained have improved mechanical and electrical properties. A. R. Powell.

Process for treating latex and products obtained thereby. S. M. Cadwell, Assr. to Naugatuck Chemical Co. (U.S.P. 1,641,573, 6.9.27. Appl., 22.9.25).—Rubber latex is mixed with a combination of substances capable of effecting accelerated vulcanisation; one of these ingredients is inactivated while in the latex, but the rubber obtained by the desiccation of the mixture is capable of vulcanisation at ordinary temperatures. D. F. Twiss.

Process of reclaiming rubber. G. J. Miller (U.S.P. 1,641,598, 6.9.27. Appl., 22.12.26).—Comminuted scrap vulcanised rubber is heated in kerosene oil at 120—190° until dissolved; after separation of solid impurities the rubber is precipitated from the cool solution by the addition of alcohol or other precipitant, a third mutually miscible liquid also being introduced. The deposit is purified by re-dissolution, *e.g.*, in carbon disulphide, and re-precipitation; it is then dried. D. F. Twiss.

Method and apparatus for vulcanising rubber articles. L. A. Laursen (E.P. 276,430—1, 27.5.26).

XV.—LEATHER; GLUE.

Neutral salt effect in chrome tanning. I. Action of neutral chlorides on the tanning property of chromic chlorides. K. H. Gustavson (Ind. Eng. Chem., 1927, **19**, 1015—1020).—In chrome tanning leather by the one-bath method the solution of basic chromium sulphate or chloride used has p_H 2·5—3·5. Sodium sulphate or chloride is added to reduce the swelling of the skin and retard the initial chrome fixation. This increases hydrogen-ion concentration in most cases, but retardation of tanning also occurs when p_H is decreased. Experiments were carried out to determine the effect of sodium chloride on the tanning property of chromium chlorides, measured by their chrome fixation by hydrated hide powder. With moderately concentrated solutions containing 7—9 g./litre Cr_2O_3 as used in practice, the addition of sodium chloride gave a progressive increase in the chrome fixation except with very basic solutions, when a reversal took place at higher sodium chloride concentrations. Further tests showed that the more dilute the chrome solution the greater was the accelerating effect of adding sodium chloride; with very concentrated solutions (100 g./litre Cr_2O_3) the addition caused a retardation. Other chlorides caused an increased effect per mol. used, calcium being the highest. Hide powder peptised by potassium thiocyanate showed a greater affinity corresponding to the change in the degree of colloidality of chromic chloride. It is recommended that tanning should begin with a moderately acid basic chloride containing a small amount of salt to prevent swelling. After a short pretannage, salt is added to give a solution of molal strength 1, greatly accelerating the rate of tanning. Lastly, alkali should be added. The important factor in the neutral salt effect in tanning is the change in the constitution of the chromic chloride complex, and not variation in p_H. C. Irwin.

Hygroscopic properties of leathers according to Wilson, Daub, and Kern's experiments. M. Hirsch (Collegium, 1927, 403—407; cf. B., 1926, 798).—The wide variations in the properties of moist and dry leathers found by Wilson, Daub, and Kern need not necessarily be due to fundamental differences, but to variations in preliminary treatment. Precautions should be taken

to prevent variations in temperature and to ensure that there are no differences in the samples. A thin chrome calf and a thick chrome side leather were exposed to a saturated atmosphere for 14 days. The chrome calf increased in area more and at a greater rate than the side. When subsequently exposed to dry air, the chrome calf diminished in area at a greater rate; this was partially due to the greater increase previously.

D. Woodroffe.

Technical production of tannin. F. Chemnitius (J. pr. Chem., 1927, [ii], **117**, 83—96).—A complete outline of the technical production of tannin by a continuous process. E. W. Wignall.

Photo-gelatin. V. Isajevič (Kolloid-Z., 1927, 42, 339—350).—A study was made of 30 characteristic properties of the sol and gel of 10 different kinds of gelatin. The photographic quality of gelatin depends largely on the presence of products of degradation. The soft kinds of gelatin contain more decomposition products than the hard kinds, are more readily dialysable, and have less tendency to form a gel. The viscosity and mol. wt. of gelatin depend on the temperature, concentration, and age of the sol. Successive cooling and warming also brings about an alteration in the size of the particles, and these changes are responsible for the indefinite m.p. of gelatin. Hard gelatin retains water and foreign matter more firmly than soft gelatin, and is more resistant to electrodialysis. A gel readily separates out from a sol of hard gelatin on electrodialysis. E. S. Hedges.

Errata. B., Sept. 9, 1927, 685, col. 1, first abstract: line 19, for "basic" read "direct cotton"; lines 22—23, for "Chrome leather dyes (direct)" read "Alizarin dyes"; line 24, for "Acid dyes" read "Sulphur dyes."

Patents.

Production of transparent parchment from animal hides. F. Znidarič (Austr. P. 105,359, 19.1.26).—The hides are treated first in a bath consisting of a mixture of sodium sulphide, sodium chloride, and calcium hydroxide, and then in another of dilute sulphuric acid, a very transparent uniform parchment being produced from the raw hide within 1 hr. Thus the washed hide is placed in the first bath for ¼ hr., taken out, scraped if necessary, placed in dilute sulphuric acid for about 10 min. with stirring, stretched on frames, and dried. The hide swells in such a way that in the splitting process it is separated into entirely uniform sheets of parchment.

B. P. Ridge.

Production of felting from non-felting animal hairs. S. Marian (Austr. P. 103,897, 18.6.24).—The hairs on the skin are treated (1) with a free acid not containing aluminium salt solution or (2) with aluminium salt solutions comprising compounds of aluminium with inorganic or organic acids, including the alums. *E.g.*, hare or rabbit skins are treated by means of a stiff brush with aluminium chloride solutions containing 30—35 g. of Al_2O_3 per litre, the solution being rubbed in to at least half the hair length. The skin is then dried, the hairs being then cut off and further treated. The product has an increased capacity for felting and a high hair strength owing to the absence of excess of acid in the caustic solution. B. P. Ridge.

Production of hair felt. I. G. Farbenind. A.-G. (G.P. 440,401, 10.4.25).—The hairs are treated with metallic salt solutions (except mercury salts), with the addition of wetting agents, especially aralkyl- or alkyl-naphthalenesulphonic acids. As metallic salts, nitrates or sulphates of iron, antimony, potassium, sodium, tin, etc. are used. B. P. Ridge.

Manufacture of plastic masses consisting of hardened casein. H. A. Cope. From Soc. Ind. des Matières Plastiques (E.P. 276,542, 1.12.26).—Powdered casein is mixed with aqueous formaldehyde, alcohol, and sodium hydroxide, and, when worked and pressed hot, forms a homogeneous, non-swelling, artificial horn block of any desired thickness. Any form of formaldehyde and any alcohol may be used; pigments or soluble dyes may be added. The alkali swelling agent may be replaced by other alkalis, carbonates, phosphates, borates, or fluorides. C. Hollins.

XVI.—AGRICULTURE.

Plant growth in acid soils. H. Haastert (Z. Pflanz. Düng., 1927, **5A**, 265—314).—Numerous plant experiments show that the sensitiveness of plants to hydrolytic and exchange acidity varies considerably, and a general classification is made. Soils treated with increasing amounts of lime indicated that the first lime additions produced proportionally the greatest reductions in both exchange and hydrolytic acidity, although the amount of lime necessary entirely to eliminate the latter was relatively very great. Many plants (notably sugar-beet) increased soil acidity during growth, and even on neutral soils liming proved beneficial. The use of physiologically acid or alkaline fertilisers affects the intensity of both hydrolytic and exchange acidity. Measurements of the p_H value of soils is not a reliable means of determining their lime requirement. The efficiency of chalk in removing acidity depends on its fineness of division. The rate of decomposition of chalk in acid soils depends mainly on the extent of the exchange acidity, but the total amount which can be decomposed is more dependent upon the hydrolytic acidity. The rate of decomposition of chalk increases with the temperature of the soil and varies with the moisture content, with maximum values when the moisture is 25—60% of the total capacity. A. G. Pollard.

Effect of varying concentrations of ammonia on the nitrifying power of the soil. D. V. Bal (Agric. J. India, 1927, **22**, 298—300).—The process of nitrification is not materially affected by concentrations up to 60 mg. of ammoniacal nitrogen per 100 g. of soil, but it is almost entirely inhibited in the presence of more than 100 mg. per 100 g. of soil. C. T. Gimingham.

Current mineral nutrient content of the plant solution as a possible means of chemical control of optimum fertilisation. B. E. Gilbert and L. J. Hardin (J. Agric. Res., 1927, **35**, 185—192).—The current mineral nutrient content of plant juices can be correlated directly with the addition of fertiliser salts. Among the principal nutrients, variations in concentration in the plant solution during growth were least in the case of potassium, greatest for nitrates, and intermediate for phosphates. It is suggested that the con-

centration of nutrients in the plant solution is indicative of the fertiliser requirement of soils. A. G. POLLARD.

Why applications of nitrogen to land may cause either an increase or decrease in the protein content of wheat. W. F. GERICKE (J. Agric. Res., 1927, **35**, 133—139).—The protein content of wheat is affected by the amount of nitrogen available to the plant at various stages of its growth. The effect of fertiliser treatment at specific stages of growth differs with different varieties of wheat. The importance of these considerations in experimental work in this connexion is emphasised. A. G. POLLARD.

Micro-method for the determination of nitrate in soil solutions and extracts. C. F. FLINT (J.S.C.I., 1927, **46**, 379—381 T).—The phenolsulphonic acid colorimetric method for the determination of nitrate in soil solutions is somewhat tedious and not sufficiently accurate. The working details of a method due to Rogozinsky are examined, and finally a micro-adaptation of the Devarda reduction method is described, which is quicker and more accurate than the phenolsulphonic acid method, and capable of handling similarly small quantities of material. A slightly modified form of the micro-ammonia still of Parnas and Wagner is employed, the Hopkins still-head being loosely packed with glass-wool with a somewhat tighter plug below to trap the alkaline spray. A few c.c. of the test liquid, which may conveniently contain from 0·2 to 6 mg. of NO_3, are placed in the still with 3 c.c. of 30% sodium hydroxide and 0·25 g. of powdered Devarda's alloy. An acid trap of 0·01*N*-sulphuric acid is arranged, and after steam heating until the vigorous reaction subsides somewhat, 20 c.c. are distilled over in 4 min. The excess acid is titrated with 0·01*N*-soda.

Contact insecticides. F. TATTERSFIELD and C. T. GIMINGHAM (J.S.C.I., 1927, **46**, 368—372 T).—In addition to a brief summary of work already noted (cf. B., 1927, 86, 87), the relative toxicities of certain *N*-heterocyclic compounds, and of the fatty acids and their salts and methyl esters, to the beam aphis are briefly discussed. In the case of the fatty acids, an attempt is made to correlate certain physical properties with toxicity.

Physical properties of commercial dusting and spraying materials. L. R. STREETER (New York Agr. Expt. Sta. Bull. 125, 1927. 12 pp.).—Sieving tests for grading arsenical and sulphur dusts have proved unreliable, owing to aggregation of the particles on the sieves. This is due, in part at least, to the development of an electric charge on the particles. Mixed dusts containing sulphur and lead arsenate are, however, electrically neutral, and do not pack on the sieves. Accurate results are obtained in grading these dusts if, before sieving, they are mixed in the proportion of 1 pt. of lead arsenate to 6 pts. of sulphur. Samples of lead arsenate may also be satisfactorily compared by suspension tests in 0·3% gelatin solution.

C. T. GIMINGHAM.

PATENTS.

Production of a mixed manure containing phosphoric acid and nitrogen. F. G. LILJENROTH (E.P. 275,843, 26.11.26. Conv., 13.8.26).—Mineral phosphate is dissolved in an amount of sulphuric acid sufficient to bring all the phosphate into solution, the calcium sulphate formed being separated and washed. The phosphoric acid is saturated with ammonia, and the calcium sulphate treated with ammonia and carbon dioxide, giving calcium carbonate and a solution of ammonium sulphate. Finally, the solutions of ammonium phosphate and ammonium sulphate are mixed and evaporated to dryness. The carbon dioxide used in the process may be recovered from the calcium carbonate formed. C. T. GIMINGHAM.

Weed-killer. CHEM. FAB. L. MEYER (G.P. 441,213, 22.1.25).—A 1·5—7% solution of salts of certain heavy metals, particularly copper nitrate, is an effective weed-killer. Copper nitrate is superior to other salts such as the sulphates of iron or copper, as it not only destroys hedge mustard while in full bloom, but also acts as a fertiliser. F. R. ENNOS.

Insecticide and disinfectant. H. LAINE (F.P. 613,046, 6.3.26).—A saturated solution of hydrocyanic acid in water, alcohol, acetone, etc. may be applied as an insecticide to plants by any suitable means, as it leaves no toxic or corrosive deposit. The solutions may also be used as a source of gaseous hydrocyanic acid, by heating at 70—80°, for the disinfection of rooms etc. F. R. ENNOS.

Manufacture of fertilisers. I. G. FARBENIND. A.-G., Assees. of C. BOSCH (U.S.P. 1,639,585, 16.8.27. Appl., 9.7.20. Conv., 29.7.16).—See E.P. 145,036; B., 1921, 443 A.

[Electrical] treatment of seeds to augment their cultural value. A. C. BENNETT (E.P. 268,291, 28.12.26. Conv., 26.3.26).

XVIII.—FERMENTATION INDUSTRIES.

Detection of fruit wine in grape wine. T. RÖTTGEN (Chem.-Ztg., 1927, **51**, 697—698).—In the method previously described (B., 1927, 24) more certain results are obtained if 18 c.c. of wine are mixed with 2 c.c. of the reagent; the green colour due to the presence of fruit wine is then more clearly seen. An alternative method of detecting fruit wine in white grape wine consists in shaking 10 c.c. of the wine with 10 c.c. of a cold saturated solution of barium hydroxide in 3% hydrogen peroxide solution. Pure grape wine produces a dirty yellow precipitate, whereas adulterated wine yields a chocolate-brown precipitate. (Cf. Müller, Vogt, and Raesch, B., 1927, 665; and Kalberer, *ibid.*, 686.)

A. R. POWELL.

PATENTS.

Production of dry yeast. C. B. HILL and M. H. GIVENS, Assrs. to NORTHWESTERN YEAST CO. (U.S.P. 1,641,676—7, 6.9.27. Appl., [A], 4.3.26, [B], 25.5.26).—(A) Yeast is grown on a mixture of predetermined quantities of ground cereals, a yeast-nourishing inorganic salt (ammonium sulphate, calcium sulphate, and calcium phosphate), and malt. The action of the yeast is stopped before the inorganic salt and the malt are consumed, so that the yeast product, the moisture content of which should be less than 14%, contains the cereals, together with the unconsumed inorganic salts and their derivatives,

all of which are available for feeding the yeast when it is used in leavening operations. (B) A quantity of malt is used in excess of that required to mash the cereals.
F. R. ENNOS.

XIX.—FOODS.

Fat content of milk from Flemish cows. M. PAGET (Ann. Chim. analyt., 1927, [ii], 9, 265—266).—The average fat content is 37·4 g./litre. The minimum yield of butter should not be less than 33—35 g./litre for mixtures of milk from different cows, and 27 g./litre for milk from individual cows, provided all the milkings of the day are mixed. F. R. ENNOS.

Polarimetric determination of sucrose in sweetened condensed milk. H. D. RICHMOND (Analyst, 1927, **52**, 525—526).—The factor 1·11 used by Revis and Payne and by Honegger (B., 1926, 992) for converting the weight of fat to volume in the calculation for the polarimetric determination of sucrose in sweetened condensed milk is considered to be erroneous. The seasonal variation of the error is here regarded as due to the condensation of a layer of serum round the fat globules; the extent of the layer will vary inversely as the temperature. Sufficient time must therefore be allowed for the sugar in the condensed layer to diffuse through the solution, so that the diluted milk should be kept for at least 30 min. at 60°, then cooled to 10°, and the acid mercuric nitrate added. Inversion should be carried out at as low a temperature as possible, Harrison's method and formula used, and the final filtrate treated with phosphotungstic acid, since mercuric nitrate is not a complete precipitant of proteins.
D. G. HEWER.

Spontaneous souring of milk in the electric field and in thunderstorms. G. H. LEOPOLD (Chem. Weekblad, 1927, **24**, 438—439).—No differences could be observed either between one series of samples of milk preserved under definite conditions of time and temperature, and another series subjected to various potential differences, both constant and alternating, but otherwise kept under the same conditions, or in the movements of bacteria (*proteus* and *paratyphus*) under the microscope when subjected to the influence of similar electric fields.
S. I. LEVY.

Determination of milk fat in milk chocolate by means of a modified xylene number. C. A. GREENLEAF (J. Assoc. Off. Agric. Chem., 1927, **10**, 396—404).—The modified xylene number for fats is the number of c.c. of 0·1*N*-alkali required to neutralise the volatile fatty acids obtained from 6·4 g. of fat, which are neither precipitated as insoluble salts by magnesium sulphate nor extracted from an acidified aqueous solution by 20% of its volume of xylene. For the determination, 10 g. of fat are saponified with 20 c.c. of glycerin–potassium hydroxide mixture, hot water and hot magnesium sulphate solution are added, the whole is heated at 80° for 5 min., cooled to 20°, bulked to 260 g., and filtered. 200 c.c. of the filtrate are treated with 50 c.c. of dilute sulphuric acid and shaken thoroughly with 50 c.c. of xylene. 200 c.c. of the filtered aqueous layer are diluted with 50 c.c. of water, exactly 200 c.c. are distilled over, and the distillate is titrated with alkali. A blank experiment is carried out at the same time. Butter fat and cacao butter give average values for the modified xylene number of 26·18 and 0·19 respectively. The graphical relationship between the values for mixtures of butter fat and cacao butter and their content of butter fat can be approximated by two straight lines, one for values from 0—40% and the other from 40—100% of butter fat. Equations are given for calculating the amount of butter fat in mixtures with cacao butter, and also in milk chocolate after extraction of the mixed fats. F. R. ENNOS.

Protein content of wheat. GERICKE.—See XVI.

PATENTS.

Process for making food products. N. M. CREGOR, Assr. to WARD BAKING CO. (U.S.P. 1,640,182, 23.8.27. Appl., 20.8.24).—The raw material consisting of cereal germs is cooked until the starch present is gelatinised, after which it is treated to effect saccharification of the gelatinished starch, boiled, and subjected to a second saccharification treatment. F. R. ENNOS.

Preservation of eggs or egg products. W. MATZKA (E.P. 276,132, 6.8.26).—The yolk and white are separated from the shell, mixed with 2—4% of common salt, and 30—45% of the water removed by evaporation *in vacuo* at a temperature low enough to prevent coagulation of the white. The concentrate is then sterilised by passing it between electrodes of aluminium and of a noble metal, *e.g.*, gold or a metal coated or tipped with gold, which are heated at the same or different temperatures below 60°, and which may also be electrically connected outside the liquid or supplied with a small current, the aluminium forming the anode. F. R. ENNOS.

Food product. A. K. BALLS (U.S.P. 1,642,320, 13.9.27. Appl., 9.11.25).—Yeast is heated at not above 150° for less than 90 min. and the resulting liquid concentrated. F. R. ENNOS.

Preservation of fruit juices and other liquids. W. MATZKA (E.P. 276,254, 1.7.26. Addn. to E.P. 267,058; B., 1927, 376).—The process of the prior patent is modified by replacing the gold by other noble metals, *e.g.*, platinum, or by copper or silver, and omitting the conductive electrical connexion between the two heated metals, so that the liquid itself acts as the conductive means for any electrical currents produced. F. R. ENNOS.

Method of treating coffee. R. CROSS (U.S.P. 1,640,648, 30.8.27. Appl., 2.11.25).—The coffee passes slowly down a treating tower where it is soaked in an alkaline solution and subjected to a current of heated inert gas, which then passes to a sublimating chamber to condense the caffeine, and to a condenser and separator to remove water and volatile oils, the uncondensed material being finally recirculated through the coffee undergoing treatment. F. R. ENNOS.

Drying apparatus [for root-crops etc.]. B. J. OWEN (E.P. 275,760, 21.6.26).—A drying apparatus suitable for materials liable to be injured by excessive heat consists of one or more vertical chambers down which the material to be treated descends continuously or intermittently. It is dried in successive stages by heated air which passes repeatedly across the chamber through different portions, and is re-heated before traversing the less dried material near the top. The sides of

the chamber are gradually sloped outward from the upper end, so that the rate of subsidence in the upper portion is increased in relation to the extent to which the material shrinks on drying. The air passages may be formed by means of strips arranged at intervals at the sides of the chamber like the slats of a Venetian blind so as to prevent clogging; suitable rods may be fixed to prevent the material falling more rapidly in the centre than at the sides and to promote mixing.

C. T. Gimingham.

Manufacture of nitrogen-containing products. M. Kahn, Assr. to Soc. Franç. des Prod. Alimentaires (U.S.P. 1,642,209, 13.9.27. Appl., 26.3.25. Conv., 1.4.24).—See E.P. 231,864; B., 1925, 939.

Pasteurisation (Austr. P. 105,348).—See I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Wormseed oil. H. Thoms (Pharm. Ztg., 1927, 72, 1123—1124).—For the determination of the ascaridol content and for the detection of falsification of wormseed oil, the German Pharmacopœia VI method is the most reliable. Unsuccessful attempts to find a more satisfactory method are described.

E. H. Sharples.

Mercaptans and petroleum distillation. Morrell and Faragher.—See II.

Rubber hydrocarbons. Bruson and others.—See XIV.

Patents.

Manufacture of cyclic hydrocarbons and derivatives thereof. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 253,911, 15.6.26. Conv., 22.6.25. Addn. to E.P. 251,270; B., 1927, 742).—The process of the prior patent is extended to the secondary alcohols (or their esters) corresponding with the ketones there used. Secondary reactions due to the hydrogen liberated are avoided by addition of air or an oxidant to the alcohol vapour, and an oxidation catalyst may be added to the porous contact mass. Phenyl-*m*-xylylcarbinol, b.p. 330°, passed with air over active carbon at 360—380°, gives 35% of 2-methylanthracene, m.p. 201°; the acetate gives the same product at 400°; di-*p*-xylylcarbinol, m.p. 131°, passed with air over active carbon charged with oxides of copper and cobalt at 400°, gives 50% of 1 : 4 : 6-trimethylanthracene, m.p. 226°. In place of the carbinols corresponding diarylmethanes may be used with excess of air.

C. Hollins.

Manufacture of aldehydes. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 267,925, 14.3.27).—The vapour of a carboxylic acid is passed with carbon monoxide (or gases containing it) over a hydrogenating catalyst (*e.g.*, iron, cobalt and iron, chromic and iron oxides, or a mixture of cerium, chromium, and iron) at 250—500°. Carbon dioxide and the corresponding aldehyde are produced. Examples are benzaldehyde and acetaldehyde from benzoic and acetic acids, respectively.

C. Hollins.

Production of methyl alcohol and other oxygenated organic compounds. Synthetic Ammonia & Nitrates, Ltd., and H. G. Smith (E.P. 275,345, 12.5.26).—By the action of slightly more than 1 mol. of aqueous chromic acid on 4 mols. of a suspension of finely-divided zinc oxide or hydroxide, there is formed the compound $4ZnO,CrO_3$. This is particularly active as a catalyst in the production of methyl alcohol from mixtures of hydrogen and carbon monoxide at high pressure, giving high yields at 300—400°. If alkali salts are intimately mixed with the catalyst, or if the latter is prepared by double decomposition of suitable salts, preferably in the presence of alkali, it becomes suitable for the production of higher alcohols and other oxygenated compounds, *e.g.*, butyl alcohol.

B. Fullman.

Manufacture of organic compounds containing oxygen. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 275,284, 4.3.26).—Compounds of a higher order than methyl alcohol, *e.g.*, *iso*butyl alcohol etc., are formed when a mixture of carbon monoxide and hydrogen (carbon dioxide being eliminated or limited to not more than 3%) is passed at high pressure and temperature over a heated contact mass. The carbon dioxide is removed by washing with water or by other means. The carbon monoxide may be partially replaced by alcohols (ethyl alcohol etc.), and the hydrogen completely or partially by alcohols or hydrocarbons, especially unsaturated or readily decomposable hydrocarbons.

B. Fullman.

Manufacture of synthetic camphor. L. and E. Darrasse and L. Dupont (E.P. 253,542, 10.6.26. Conv., 12.6.25).—In modification of E.P. 164,357 (B., 1922, 610 A), the treatment of turpentine with oxalic acid is carried out in the presence, as solvents, of benzene, toluene, etc. or their mixtures to the amount of 2 or 3 times the weight of the turpentine.

B. Fullman.

Manufacture of substituted aromatic sulphonic acids. I. G. Farbenind. A.-G. (E.P. 269,155, 24.3.27. Conv., 7.4.26. Addn. to E.P. 253,188; B., 1927, 618).—In modification of the main patent methyl or ethyl alcohol is added simultaneously with chlorosulphonic acid and/or oleum to an aromatic hydrocarbon, acid and alcohol being separate or mixed; or the alcohol is added to a mixture of acid and hydrocarbon.

B. Fullman.

Manufacture of derivatives of cinchona alkaloids. C. F. Boehringer & Soehne, G.m.b.H., Assees. of A. Rothmann and V. Hilcken (G.P. 439,466, 9.9.24).—Cinchona alkaloids are treated with a cyanogen halide and then with alkali or with hydrogen cyanide to give therapeutically valuable *N*-cyano- and *N*-dicyano-derivatives. *Quinine bromocyanide*, m.p. 94°, is converted by ammonia solution into *cyanoquinine*, $C_{21}H_{24}O_2N_3$, m.p. 225—226° (*hydrochloride* described). Hydroquinine gives a *bromocyanide*, m.p. 165° (decomp.), and *cyanohydroquinine*, $C_{21}H_{26}O_2N_3$, m.p. 223° (*hydrochloride*). Other compounds described are: *cinchonidine bromocyanide*, *cyanocinchonidine*, m.p. 195° (*hydrochloride*), *hydrocupreïne bromocyanide*, *cyanohydrocupreïne*, m.p. 215° (decomp.; *hydrochloride*). Hydroquinine bromocyanide combines with hydrogen cyanide in dry chloroform to give *dicyanohydroquinine hydrobromide*, $C_{22}H_{27}O_2N_4Br$, m.p. 202—204°.

C. Hollins.

Extraction of phosphatides from the soya bean. J. D. RIEDEL A.-G. (G.P. 439,387, 27.5.23).—The oil-free phosphatides obtained by extraction of soya beans with benzene–alcohol are treated with alcohol to remove lecithin, dissolved in an organic solvent (benzene, methyl*cyclo*hexane, chloroform, or carbonate trachloride), filtered from impurities, and pure phosphatides, containing phosphorus and nitrogen in atomic proportions, are precipitated by addition of alcohol or acetone. The products give in water stable colloidal solutions precipitated by metallic salts, and on hydrolysis yield glycerophosphoric acids, aliphatic acids, and organic bases in similar ratio as in the case of lecithin. C. HOLLINS.

Production of iodised pyridine derivatives. C. RÄTH (E.P. 252,707, 17.5.26. Conv., 28.5.25).—In modification of E.P. 251,578 (B., 1926, 608), 3- or 5-iodopyridine derivatives are prepared from the corresponding amines (such as 5-amino-2-hydroxypyridine) by treatment of the latter with iodides in the presence of an acid solvent and subsequent addition of alkali nitrite, with or without the addition of catalysts such as copper. The preparation of 5-iodo-2-hydroxypyridine, m.p. 191—192°, is described. B. FULLMAN.

Production of organic arseno-compounds. A. BINZ and C. RÄTH (E.P. 255,839 and 255,861, [A] 30.6.26, [B] 15.7.26. Conv., [A], 22.7.25).—Arseno-compounds are obtained in increased yield when the condensation of an arsine (A) with an arsenoxide, with a dihalogenoarsine, or (B) with the unisolated reduction product of an arsinic acid, is conducted in presence of small quantities of a reducing agent such as hypophosphorous acid or its salts (cf. E.P. 11,901 of 1911, and E.P. 250,577; B., 1912, 256; 1927, 670). C. HOLLINS.

Manufacture of a compound of *iso*propylallylbarbituric acid with antipyrine. F. HOFFMANN–LA ROCHE & Co. A.-G. (Swiss P. 116,465, 9.5.25).—The components in equimolecular proportions are dissolved in alcohol, acetone, ether, benzene, chloroform, or water, and the *compound*, m.p. 94°, is allowed to crystallise out. It is more soluble than either component and a stronger antipyretic and analgesic than antipyrine. C. HOLLINS.

Process for oxidising organic compounds. SILESIA VER. CHEM. FABR. (E.P. 259,930, 10.9.26. Conv., 19.10.25).—Organic compounds are oxidised by polythionates in alkaline solution. Trithionates are converted into thiosulphates and sulphites according to the equation: $S(SO_3Na)_2 + 2NaOH = NaS{\cdot}SO_3Na + Na{\cdot}SO_3Na + 2OH$. Tetrathionates give 2 mols. of thiosulphate, whilst pentathionates give 2 mols. of thiosulphate and an atom of sulphur. The reactions are particularly suitable for the oxidation of dithiocarbamates to thiuram disulphides. C. HOLLINS.

Obtaining sulpho-aromatic fatty acids. G. PETROFF and P. SHESTAKOFF (U.S.P. 1,642,595, 13.9.27. Appl., 30.12.25. Conv., 18.5.25).—See E.P. 252,212; B., 1927, 348.

Quinoline derivatives (E.P. 276,156).—See IV.

Salts of *o*-aminophenylpropionic acid etc. (F.P. 613,596).—See IV.

Vulcanisation accelerators (E.P. 276,435).—See XIV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photo-gelatin. ISAJEVIČ. See XV.

PATENTS.

Sensitised coatings and processes for photo-etching. J. RIEDER (E.P. 275,763, 29.6.26).—The surface to be etched is coated with a layer of varnish consisting mainly of resin, such as sandarac, dragons' blood, or shellac, which is insoluble in petroleum, benzine, and similar solvents, together with just sufficient benzine-soluble, sensitive asphalt, with or without rubber, to render the mixture soluble in benzine, petroleum, etc. The exposure is considerably shortened, in that it is only required to render insoluble a small amount of asphalt. A slightly higher asphalt content may be added to the sensitive coating, if a paraffin oil, stearine oil, etc. is added to the developer to reduce the solvent property. The designs may be printed directly on the sensitive coating, or indirectly using a coloured image which on the one hand impedes the light, and on the other reduces the sensitiveness of the layer. W. CLARK.

Protecting the gelatin coating of photographic plates and films. C. GLASER (U.S.P. 1,640,148, 23.8.27. Appl., 7.10.26. Conv., 14.6.26).—The plates are coated with a mixture of 300 g. of a solution of transparent celluloid, 150 g. of gum-lac solution, 450 g. of ethyl acetate, and 150 g. of glacial acetic acid. The celluloid solution is composed of 85—100 g. of plastic material dissolved in 800—850 g. of ethyl acetate, mixed with 35—50 g. of amyl acetate; the solution of gum-lac consists of 200—250 g. of gum-lac dissolved in 700—750 g. of alcohol at 96°. W. CLARK.

Recovery of values [camphor] from [photographic] nitrocellulose material. C. ELLIS and H. M. WEBER, Assrs. to ELLIS-FOSTER Co. (U.S.P. 1,637,990, 2.8.27. Appl., 9.5.21).—Waste photographic film is slowly added to warm aqueous sodium hydroxide (20%), the solution obtained being freed from camphor by extraction with benzene, and filtered from silver compounds. T. S. WHEELER.

Photographic process. G. ROUSSEAU (F.P. 608,391, 24.12.25).—Of three superimposed films, the upper carries a normal emulsion, the next an emulsion sensitised to green and yellow, and the last a red-sensitised emulsion. Between the upper and middle films a thin Auramine film may be interposed, and between the middle and lower films a Fuchsin film. The three films are used just like an ordinary photographic plate. W. CLARK.

Photographic process and papers. Y. M. LETORT and R. C. F. BORDE (F.P. 611,670 and 611,672, 9.2.26).—With the view of decreasing the time of exposure, the sensitive coating is mixed with or coated with phosphorescent substances. Photographic papers are coated with a layer of phosphorescent salt and then with the emulsion. Prints made from such a paper phosphoresce in the dark. W. CLARK.

Process of colour photography. Y. M. LETORT and R. C. F. BORDE (F.P. 611,671, 9.2.26).—Portions of a silver bromide-gelatin emulsion are dyed orange, green, and red-violet with dyes which are stable in the developer. The portions of emulsion are then finely powdered, the powders are intimately mixed, and the mixture is coated on the support (glass plates). Suitable substances are mixed with diazo compounds which become blue, green, or red-violet under the action of light, and with light-stable orange, green, or red-violet dyes and the mass is finely powdered or else the powder containing the diazo compounds is mixed separately with the stable dyestuffs. Gelatin dyed red-violet, green, or orange and powdered and coated on paper may be sensitised with dichromate. W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Quantitative stability test for smokeless powders. J. C. A. S. THOMAS (Z. angew. Chem., 1927, **40**, 991—992).—The powders are heated in an oil-bath, maintained at 104—106° for cordites and 109—111° for nitrocellulose, for 8 hrs. and weighed. The powders so dried are then heated again in the same bath for various periods up to 120 hrs., being weighed morning and evening. Satisfactory powders should not lose more than 2% of the dry weight in 72 hrs. The results obtained with a wide range of powders in which different stabilisers had been employed in varying quantities are given. Whilst cordite does not pass the test unless a stabiliser has been used, guncotton properly prepared is stable without addition. S. I. LEVY.

Gaseous products of explosion of blasting explosives. J. THORBURN (J.S.C.I., 1927, **46**, 358—361 T).—A chamber of 72 cub. ft. capacity, made from $\frac{3}{8}$ in. mild steel plate with a manhole 2 ft. 4 in. in diam. of $\frac{1}{2}$ in. plate, was designed to withstand the explosive effect of a 1-lb. charge of an explosive of average strength fired at the centre of the closed vessel. The vessel, strengthened with reinforced concrete, is sunk in the ground. Shots may be fired unconfined or confined, in air or in nitrogen, and at normal or reduced pressures. The charge is fired electrically, and a sample of the gaseous products is immediately drawn into evacuated receptacles for the determination of nitrous fumes, free ammonia, and carbon dioxide. A second sample is then drawn into evacuated pipettes for the determination of carbon monoxide, hydrogen, and methane and checking the carbon dioxide. Some of the results obtained on firing various blasting explosives in whinstone blocks suspended in the chamber are given.

Determination of carbon monoxide, hydrogen, and methane in air containing ethylene. THORBURN.—See II.

PATENT.

Production of explosives. F. H. BERGEIM, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,637,726, 2.8.27. Appl., 18.6.26).—Ethylene glycol containing ethylene oxide (2—50%) is nitrated with a mixture (2 : 3) of nitric and sulphuric acids, to yield a neutral stable oil of value as a substitute for nitroglycerin. T. S. WHEELER.

XXIII.—SANITATION; WATER PURIFICATION.

Water analysis. W. R. ATKIN and D. BURTON (J. Soc. Leather Trades' Chem., 1927, **11**, 294—295).—To avoid complications, bromophenol blue is used as indicator in all titrations in the determinations of temporary, permanent, and magnesia hardness, respectively (cf. J.S.C.I., 1927, **46**, 111—114 T; B., 1927, 382). D. WOODROFFE.

Fate of grease in sludge digestion. S. L. NEAVE and A. M. BUSWELL (Ind. Eng. Chem., 1927, **19**, 1012—1014).—Rapid destruction of grease and calcium soaps with production of lower fatty acids occurs in the acid type of sludge digestion. The rate of digestion, but not the extent, appears to be increased by the addition of lime (cf. B., 1927, 269), which addition also produces peaks in the curve for gas evolution and for production of lower fatty acids. In the former case, however, increased liquefaction of solid matter is not necessarily indicated, since the gas originates from substances already in solution; in the latter case, the peak may merely indicate the formation of soluble or peptised calcium salts. The methane produced comes from fermentation of the lower fatty acids, and not from degradation of cellulose present, the latter undergoing little, if any, change during the ordinary sludge-digestion period. The small amount of hydrogen produced early in digestion is probably an approximate indication of the quantity of available carbohydrates, since they usually ferment anaerobically to carbon dioxide and hydrogen. Thus, when seeded with sludge liquor, inulin, starch, dextrin, raffinose, maltose, sucrose, lactose, salicin, dextrose, lævulose, dulcitol, mannitol, adonitol, and glycerol all afford gases containing hydrogen but not methane. A p_H of 5·0 is too acid for good digestion of the sludge, proteolysis being hindered. In addition to liming and utilising the buffer value of a large excess of alkaline sludge, slight aeration appears to offer possibilities. The rate of fermentation, as measured by gas production, is roughly proportional to the grease content of the dry sludge, a scum of high grease content giving the most vigorous evolution of gas. R. BRIGHTMAN.

PATENTS.

Apparatus for the electrolytic purification of water, especially boiler-feed water. SIEMENS-SCHUCKERTWERKE G.M.B.H. (G.P. 440,979, 17.5.22).—The water is subjected to electrolytic treatment under reduced pressure. L. A. COLES.

Production of suffocating and poisonous gases for combating vermin. H. SAUER, JUN. (E.P. 250,274, 6.4.26. Conv., 3.4.25).—A cartridge containing sawdust and a combustible which is independent of the presence of air for its combustion, is burnt within the habitation of the vermin, and by incomplete or slow combustion in absence of air yields a comparatively large amount of carbon monoxide, together with other asphyxiating gases. W. G. CAREY.

Sterilisation of liquids. A. SCHREIER, Assr. to GEN. ZEOLITE Co. (U.S.P. 1,642,089, 13.9.27. Appl., 8.1.21. Conv., 8.1.20).—See E.P. 157,280; B., 1922, 116 A.

Disinfectant (F.P. 613,046).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOVEMBER 11, 1927.

I.—GENERAL; PLANT; MACHINERY.

Simple face mask for dusty industrial processes. L. P. Lockhart (J. Ind. Hyg., 1927, 9, 421—423).

Patents.

Grinding, milling, and pulverising apparatus. R. K. Ramsay, and Mayhew, Ramsay, & Co., Ltd. (E.P. 277,185, 14.10.26).—In a pulveriser of the swing-hammer type with a perforated base to the grinding chamber, a current of air is drawn from atmosphere through the mill by a fan, and the air, after depositing the ground material in a separator, is not returned to the mill, but exhausted to atmosphere.

B. M. Venables.

Grinding mills. R. Lister & Cie., and F. V. Lister (E.P. 276,594, 22.4.27).—A fine-grinding or colloid mill is constructed with the space between the rotor and stator gradually decreasing at first from the inlet outwards and then with a parallel zone.

B. M. Venables.

Crushers. Allis-Chalmers Manuf. Co., Assees. of R. C. Newhouse (E.P. 269,866, 15.3.27. U.S., 24.4.26).—Several crushers having unbalanced moving parts, *e.g.*, gyratory crushers, are geared together so that the whole is in balance. B. M. Venables.

Apparatus for heating substances in a finely-divided state. J. Trautmann (E.P. 262,791, 8.12.26. Ger., 8.12.25).—The powdered material is blown into a stream of hot gas and the mixture passes at least once upwards and once downwards through concentric passages in succession before the powder is allowed to separate out. The apparatus may be arranged to work under pressure. B. M. Venables.

Process of transforming pulverulent substances into uniform small pieces for reaction with gases. I. G. Farbenind. A.-G. (E.P. 269,209, 11.4.27. Ger., 10.4.26).—The dry powder is spread in a layer 1—2 cm. thick upon a conveyor belt, and on to it is scattered liquid in the form of individual drops, precautions being taken to prevent drops running together. Each drop of liquid will gather up powder into the form of a nearly spherical pellet, and the pellets are separated from excess powder by screening, then dried, and placed in the reaction vessel, where they will offer but little resistance to the flow of gases, but provide a large active surface. B. M. Venables.

Production of dispersion of solids in liquids and apparatus therefor. W. H. Whatmough (E.P. [A, B] 276,400—1, 22.4.26, and [C, D] 276,727—8, 29.5.26).—(A) The apparatus may conveniently be used after a colloid mill of known type in order to produce more concentrated dispersions by breaking down the non-colloidal globules that will pass through other types of mill. The paste (or fluid) is subjected to a wiping action between a cylindrical or conical rotating surface and fixed surfaces lined with yielding material such as plush. Temperature may be controlled if desired. In (B) is described an adaptation of an existing type of roller mill to effect the process described in (A). (C) In a mill having both surfaces in the form of flat discs, one disc is rotated and the same or the other is given a reciprocating straight-line or arcuate motion. (D) Both discs rotate about non-coincident axes.

B. M. Venables.

Method of thickening [filtering] mixtures. A. L. Genter, Assr. to Genter Thickener Co. (U.S.P. 1,642,673, 20.9.27. Appl., 16.7.25).—The hot mixture, in contact with a filtering medium, forms two filtrate columns united at their lower ends. Suction is applied to one column to promote the filtration, and the density of the liquid in that column is reduced to raise its level above that of the liquid in the other column.

H. Holmes.

Filtering machines. E. L. Smith. From W. T. Chatfield (E.P. 276,820, 19.8.26).—A filter especially suitable in the treatment of gold slimes etc. is constructed of a 5-sided box, the 6th or open end being provided with flanges by which the box can be bolted to a fixed end-plate with a movable frame interposed. A number of filter leaves are constructed around tubular supports attached to the fixed end-plate, and the movable frame is made up into a grid with more or less flexible cross-slats which will scrape the surfaces of the filter leaves. For filtering, the apparatus is bolted up and the prefilt supplied under pressure through the fixed end to the interior of the box. After filtering and washing, the bolts are unfastened and the box is run back on wheels and rails; at first it runs back alone, but in the latter part of its motion drags the frame with it and scrapes the filter cakes off. B. M. Venables.

Heating of liquids. A. J. Lambert, and Lambert Heater & Engineering Co., Ltd. (E.P. 276,807, 27.7.26). —A heating vessel—which may be made in sizes suitable for a domestic kettle upwards—is provided with a "bottom" that is cupped very steeply upwards, so that the upper bend is nearly at the liquid level, and the liquid is substantially all contained in the jacket between the "bottom" and the sides of the outer vessel. The surface of the "bottom," may be corrugated and may have attached to it, but spaced from it, on the fire side, rectangular or other shaped thin box-like structures containing liquid which circulates into the main body through pipe-like connexions. B. M. Venables.

Apparatus for concentrating or solidifying liquids, pastes, etc. G. H. Warburton (E.P. 276,475, 5.7.26).—The paste is passed over several rollers in succession and removed from the last by a knife or scraper. The last roller is heated by internal steam, which is produced and reproduced in a heating coil adjacent to the apparatus, and circulated by a pump driven by the apparatus. B. M. Venables.

Gas and air washing apparatus. Pneumatic Conveyance & Extraction, Ltd., and S. N. Chew (E.P. 277,112, 10.6.26).—The gas to be washed passes under a screen of flexible material, such as tarpaulin, which is attached to wooden or other bars floating on the surface of the washing liquid, and from the bars depend strips or sheets of metal broadside on and in staggered relation to the flow of gas. On its passage between the strips the gas produces waves and splashing, keeping all the surfaces wet and washing off the collected mud, which settles in the bottom. B. M. Venables.

Indicating or controlling devices operating in the presence of impurities or other additions in gases or liquids. Deuts. Gasglühlicht-Auer-Ges.m.b.H. (E.P. 254,299, 17.6.26. Ger., 27.6.25).—A brick is made of a substance that will crumble when acted on by the gas, vapour, or liquid which is to be detected, the crumbling of the brick serving to operate a warning or controlling device. For example, the presence of benzol in a current of gas or stream of liquid such as alcohol may be detected by a pellet of rubber. B. M. Venables.

Process and apparatus for mixing gases. E. Falkenthal (U.S.P. 1,643,065, 20.9.27. Appl., 18.12.24. Ger., 23.4.21).—A vessel provided with gas inlet and outlet pipes is divided by a central partition formed of thin grids spaced to constitute a mixing chamber, each grid being provided with a large number of regular openings. Opposed streams of the gases are passed slowly into the mixing chamber through the grids. The smallest diameters of the openings are about ten times the thickness of the grid, and their edges are sharp to promote a whirling motion of the gases behind each opening. H. Holmes.

Carrying out exothermic reactions. L'Air Liquide Soc. Anon. pour l'Étude et l'Exploit. des Proc. G. Claude, Assees. of Soc. Chim. de la Grande Paroisse (E.P. 268,722, 26.10.26. Fr., 1.4.26).—In a catalyser for carrying out exothermic reactions, such as the combination of nitrogen and hydrogen, the incoming gases are conveyed through the catalyst (out of contact with it) to the hottest part, then brought back to the cooler part, and finally back to the hotter part, and emerge into the catalyst. By this means the catalyst is maintained at a more uniform temperature and the gases are finally delivered into the catalyser at a temperature high enough to maintain the reaction. Part of the incoming gases (or other gases) may be passed through a narrow space between the catalyst and the containing pressure-resisting vessel to protect the latter. B. M. Venables.

Heating, melting, evaporating, or cooling vessel or the like. P. Hadamovsky (E.P. 265,597, 3.2.27. Ger., 3.2.26).—A heating or cooling element is constructed, *e.g.*, by casting iron round wrought-iron pipe coils and giving the cast iron surface a corrugated form, so that all points of the surface in contact with the liquid are equidistant from the source of heat or cold. B. M. Venables.

Tunnel kiln. J. Kelleher, Assr. to Harper Electric Furnace Corp. (U.S.P. 1,643,775, 27.9.27. Appl., 29.8.25).—Two streams of gas, substantially filling separate portions of the tunnel, enter the tunnel under pressure, and are withdrawn at adjacent points at substantially the same pressure below atmospheric, so that the gases are in contact near their points of withdrawal. J. S. G. Thomas.

Apparatus for preventing damage by gases evolved in mordanting, electrolysis, or in the production of chemical products. Soc. Anon. de Perfectionnements Electrolytiques (F.P. 617,551, 15.6.26).—Air, blown through the gap between cover plates arranged at different heights on the surface of the vessel, carries the evolved gases to a hood. J. S. G. Thomas.

Refrigeration. Carrier Engineering Co., Ltd., and W. H. Carrier (E.P. 276,887, 21.2.27).—Methylene chloride, d^{15} 1·33, b.p. 39·5—40·5°/760 mm., is subjected to compression, condensation, and evaporation in a refrigerating cycle, the pressure never being much above atmospheric and preferably always below it. The quantity of liquid in circulation is sufficient to leave some unevaporated, the total circulating liquid being returned by a pump separate from the compressor, which deals with the vapour which alone passes through the condenser. The evaporating surfaces are unsubmerged, and are covered by sprays above them. B. M. Venables.

Colour-estimating apparatus. Tintometer, Ltd., F. E. Lovibond, and G. S. Fawcett (E.P. 277,166, 3.9.26).—A tintometer is arranged for the simultaneous viewing of a standard white background through one of a series of standard colour-screens, and of a pastille or other sample of material through a blank aperture, both background and pellet being illuminated by the same source of artificial light. The pastille may be of material that changes its colour when exposed to ultraviolet light, and is exposed to the same rays as a clinical patient, so that the dosage can be measured by the change of colour. B. M. Venables.

[Porcelain] suction filters. Staatliche Porzellan-Manuf. (E.P. 264,838, 18.1.27. Ger., 19.1.26).—See G.P. 433,376 ; B., 1927, 189.

Method and agent for drying gases. W. Müller, Assr. to I. G. Farbenind. A.-G. (U.S.P. 1,644,439, 4.10.27. Appl., 30.3.27. Ger., 7.6.26).—See E.P. 272,173 ; B., 1927, 736.

Apparatus for effecting chemical tests and controlling chemical reactions. H. S. Hatfield (U.S.P. 1,643,243, 20.9.27. Appl., 9.10.26. U.K., 14.10.25).—See E.P. 264,237 ; B., 1927, 207.

Apparatus for screening fine coal or other materials. W. H. Barker (E.P. 277,392, 11.3.26).

Devices for intimately mixing, churning, or agitating liquids. R. Ruben, Ltd., and R. Ruben (E.P. 277,564, 10.3.27).

[Circulating devices for] absorption refrigerating machines. A. J. E. Munters (E.P. 255,035, 15.6.26).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Influence of the ash constituents in the carbonisation and gasification of coal. Gas Fellowship 1927 Rept. (I) J. A. SUTCLIFFE and J. W. COBB, (II) W. R. BRANSON and J. W. COBB, (III) F. J. DENT and J. W. COBB (Fuel, 1927, **6**, 449—472; cf. B., 1926, 1001—2; 1927, 592).—A further study has been made of the reactivity of the special cokes made by the carbonisation at 1000° of coal, containing originally 1% of ash, to which small amounts (up to 5%) of various oxides have been added. (I) *Gasification in steam.* Nitrogen saturated with steam was passed over a column of coke (10 g., $\frac{1}{8}$—$\frac{1}{6}$ in. size) at 1000°, the steaming rate being approximately 10 litres/hr., and the products were collected and determined. The percentages of steam decomposed by the "pure" coke and by cokes produced by the addition of 1% of ferric oxide, sodium oxide (as carbonate), and calcium oxide (as carbonate) to the coal were 62·7, 86·3, 88·4, and 74·8, respectively. No exceptional effects were produced by the addition of two oxides together. Similar increases in reactivity were observed on the addition of the same oxides to a coal containing 4·5% of ash; this had a higher original reactivity than the low-ash coal, however, and the increases were smaller. A similar increased activity towards steam was shown by these cokes at temperatures between 1000° and 600°. The reactivity of the sodium carbonate coke towards steam did not appear to decrease to any marked extent as gasification proceeded. (II) *Gasification in carbon dioxide.* Carbon dioxide at the rate of 6 litres/hr. was passed over the column of coke (10 g.) maintained at 850°, and the resulting gases were analysed. The addition of sodium carbonate to the coal produced a great increase in the reactivity of the coke as measured by the percentage of carbon dioxide reduced, but this reactivity fell off rapidly with time. The iron oxide coke was initially more active than the lime coke, but fell to a value between that for the latter and that for "pure" coke. The differences in reactivity observed at 850° become very much less marked when the determinations are carried out at higher temperatures. The increased activity due to the addition of 1% of an oxide to the coal is generally slightly more than half that due to the addition of 5%. Greatly enhanced reactivities are also produced by direct impregnation of the coke with sodium carbonate. Subsequent passage of hydrogen over the iron oxide coke at 850° restored the initial reactivity; neither the "pure" coke nor the sodium carbonate coke was affected by this treatment. (III) *Gasification in air and oxygen.* An oxygen-nitrogen mixture containing 2% of oxygen was passed over the surface of a piece of coke, 1—2 cm.^{2} in area, held in position in a tube maintained at 800°. The activity, as measured by the rate of combination with oxygen, varied very little for the various cokes, and appeared to be determined mainly by the rate of diffusion of oxygen to the surface of the coke. There are, however, wide differences in the behaviour of the cokes towards oxygen at temperatures within the range 400—700°. The iron oxide coke appears to be definitely more reactive than the "pure" coke. The sodium carbonate cokes behave abnormally, exhibiting a slow rate of combination with oxygen below 600°, and a marked increase in the rate when the temperature has passed that point. There is no connexion between the reactivities of coke towards oxygen at low temperatures and at high temperatures. Experiments on the readiness with which cokes heat up were carried out by passing a stream of air through a column of coke initially at 700°, and determining the temperature attained as a function of the time. The results indicate that in industrial operations where high temperatures are required in a bed of fuel, a coke with a low reactivity towards carbon dioxide will be more suit able than one with a high reactivity. A. B. MANNING.

Ash content of lignites. F. FISCHER and W. FUCHS (Brennstoff-Chem., 1927, **8**, 291—293).—Cassel lignite, which has been exhaustively extracted with alcohol and benzene, exhibits base-exchanging properties. The lignite contains the calcium salt of a humic acid in which, when washed with a solution of sodium, potassium, or ferric chloride, the calcium is partly or completely replaced by the corresponding metal. The calcium can be returned to the lignite by treating with a solution of calcium chloride the sample which has been washed with sodium chloride. Lignite which has been treated with hydrochloric acid takes up calcium from a solution of calcium acetate, acetic acid being found in the solution. Conversely, treatment with hydrochloric acid sets free the humic acid in the lignite and renders it much more soluble in organic solvents.
W. T. K. BRAUNHOLTZ.

Volumetric method for the determination of the ash content and the calorific value of coal. P. N. LATEGAN (Fuel, 1927, **6**, 447—448).—The ash content and the calorific value of coals deposited under similar conditions in a particular area are linear functions of the sp. gr. of the coal. When the constants of these functions are known the values of the ash content and calorific value of another coal from the same area can be deduced, with an error usually not exceeding 1%, from a determination of the sp. gr., for which the use of the Taffanel voluménomètre is recommended.
A. B. MANNING.

Determination of the reducing power of coke. G. AGDE and H. SCHMITT (Stahl u. Eisen, 1927, **47**, 1477—1481).—The apparatus comprises a source of carbon dioxide free from air and moisture, a flowmeter for measuring the rate of flow of the gas through the apparatus, drying tubes, a wire-wound electric furnace with vertical tube and regulating rheostat, a gas reservoir for collecting the issuing gases, and the usual burettes for gas analysis. The tube of the furnace is packed with grains of coke 3—4 mm. in diameter (the volume of coke used in comparative tests being kept constant) and heated at 200° for 2—3 hrs. while a current of carbon dioxide is passed through the entire apparatus with the exception of the gas burettes, so as to expel all traces of air and completely to dry the coke. The temperature is now raised to 900—1000° and the gas stream regulated to a definite rate, which is kept constant throughout a series of tests. Heating is continued for 2—3 hrs. and samples of the issuing gases are taken every 10 min. and analysed for carbon monoxide and dioxide; the

ratio $100(CO/CO_2)$ is the reducing power of the coke, the mean value of all the tests being taken. By increasing the temperature of the coke very slowly after the drying operation and observing the point at which carbon monoxide commences to be present in the issuing gases the so-called "reduction point" of the coke may be found. A. R. POWELL.

Reducing power of coke. G. AGDE and H. SCHMITT (Z. angew. Chem., 1927, **40**, 1007—1008, 1027—1032; see preceding abstract).—Experiments with synthetic coke made by carbonising a mixture of soot and pitch at various temperatures and with a coke made from a high-grade bituminous coal show that the chemical composition and physical nature, both of which depend on the temperature of formation and rate of heating, have a great influence on the reducing power. In general, the lower the temperature at which the coke is made the higher is the reducing power, owing to the greater proportion of adsorbed gases in low-temperature coke. A. R. POWELL.

Sulphur in gas coke. R. FORRIÈRES (J. Usines à Gaz; through Gas J., 1927, **180**, 42).—The sulphide sulphur in gas coke may exceed by as much as 42% the amount corresponding to the sulphate and pyrites in the original coal. This is due to interaction between the organic sulphur in the coal and iron present as oxide or other compound not containing sulphur. A further similar conversion of organic sulphur into sulphide sulphur can occur during subsequent prolonged heating of the coke. The addition of carbon dioxide to the water used for quenching coke does not increase the desulphurisation which occurs during the operation. Experiments on desulphurisation by the addition of sodium carbonate etc. to the coal, or by quenching the coke with water containing small quantities of hydrochloric acid, were inconclusive. A. B. MANNING.

Growth of fungi on coals. II. F. FISCHER and W. FUCHS (Brennstoff-Chem., 1927, **8**, 293—295).—Various types of fungi (*Penicillium*, *Aspergillus*, *Mucor*) can exist on a variety of coals. It is not essential to supply a nutrient medium, nor does air appear to be absolutely necessary to the growth of the fungi, which occur on lignite, semi-coke, and coal with diminishing readiness. Fungoid growth has also been observed on a raw Rhenish lignite. W. T. K. BRAUNHOLTZ.

Recovery of ammonia and sulphur from coal distillation gases by the Burkheiser process. W. BURKHEISER (Gas- u. Wasserfach, 1927, **70**, 943—945).—Theoretically and practically the absorption of ammonia and sulphur dioxide in the Burkheiser process is found to be quite feasible, irregularities occurring only when hydrogen sulphide is not previously completely removed from the gas. Further large-scale tests are required to determine whether the process as a whole is practicable, *e.g.*, whether carbon dioxide causes any side reactions in the saturating and regenerating stages. The ammonium sulphite, which is obtained as white crystals, loses no ammonia when stored, but is almost completely oxidised, after some days, to ammonium sulphate. W. T. K. BRAUNHOLTZ.

Determination of naphthalene in gas. J. BONTE (Bull. Soc. chim. Belg., **36**, 485—490).—An apparatus consisting of two concentric glass tubes is inserted into the flow of gas, the inner end of the outer tube being stoppered, whilst that of the inner tube is perforated. The gas enters through the side of the outer tube, is filtered in the annular space (which is packed with glass wool), and passes out through the inner tube to a condenser and finally to a vessel where its temperature is reduced to 0°. The deposited naphthalene is separated from the condensed water, dissolved in a known volume of warm acetic acid, cooled to 17°, and water added until the first traces of naphthalene are precipitated. A graph is given showing the relation between the volume of water required to produce precipitation and the quantity of naphthalene in solution.
H. D. GREENWOOD.

Determination of small quantities of carbon monoxide. D. STAVORINUS (Het Gas, 1927, **47**, 162—168; Chem. Zentr., 1927, I, 2259).—Colorimetric, titrimetric, and gravimetric methods of determining small quantities of carbon monoxide, especially by the use of ammonium chloropalladate, with or without addition of gold chloride, and also by means of iodine pentoxide, are critically discussed; even with great care none is completely reliable. The hæmoglobin method only is free from objection. A. B. MANNING.

Corrosion of silica retorts [in gas manufacture]. G. LE B. DIAMOND (Gas J., 1927, **179**, 105—107).—Jointing materials for silica retorts are not sufficiently adhesive, and from the manner in which the retorts are built up the jointing material underneath the bottom tile is peculiarly liable to fall out. This seems to occur invariably with the base-tile joint on the combustion chamber side of the retort. The horizontal section of the joint remains unaffected until it becomes fissured transversely by the longitudinal expansion and contraction of the retort. Under these circumstances producer gas leaks through the joint and deposits therein dust and fluxes, with the result that the whole zone around the joint becomes affected. To avoid this it is suggested that the base tile of the retort should be free from joints, especially on the combustion chamber side. The fact that the vertical cracks in the crown of the retort do not suffer similarly to those in the base appears to be due to the protective action of the carbon deposited in the joints from the coal gas. S. PEXTON.

Lubricating oils from coal. H. NIELSEN and S. BAKER (Brennstoff-Chem., 1927, **8**, 289—291).—The higher fraction of the tar (45% of the water-free tar, b.p. above 370°) obtained by the low-temperature carbonisation, by the "L. & N." process, of Shirebrook pseudo-cannel coal yields, after treatment with sodium hydroxide and dilute sulphuric acid, separation of paraffin, and re-distillation, a lubricating oil having lubricating properties not inferior to those of a good mineral lubricating oil. W. T. K. BRAUNHOLTZ.

Determination of water in oils. H. PFLUG (Chem.-Ztg., 1927, **51**, 717—718).—A modification of Oertel's method (cf. B., 1920, 824 A) is described in which 25 g. of the oil are mixed with 10 g. of a mixture of 2 pts. of anhydrous magnesium sulphate and 1 pt. of quartz powder in a heat-insulated apparatus of stated dimensions, and the rise of temperature is observed. For rises

of temperature up to 13° the radiation losses are practically uniform, and the percentages of water in oils of mean sp. heat 0·5 g.-cal./g. may be calculated from the observed rise of temperature by means of a constant factor 0·6. When using an apparatus of different dimensions or when examining oils with appreciably different sp. heats, other factors are applicable. In the case of oils with a moisture content greater than 8%, which give a rise of temperature of more than 13°, a preliminary dilution with a known quantity of water-free oil is necessary. F. R. Ennos.

Peat and peat wax from Chatham Islands (Bull. Imp. Inst., 25, 243—250).—The dried peat contained 25% of wax extractable with benzene and similar solvents, as compared with 6—8% from German peats and about 12% from lignite. The wax resembled crude montan wax from German lignite, but could not be readily bleached. The crude wax might form a substitute for black montan wax, and the residue, after extraction, a useful fuel. H. D. Greenwood.

Carburisation of iron. Bramley and Lawton.
Coke for blast-furnace work. Gill.—See X.

Water in alcohol-benzene mixtures. Peters.—See XX.

Water contaminated with coal gas. Regenstein.—See XXIII.

Patents.

Testing blast-furnace coke. Eisen- u. Stahlwerk Hoesch A.-G. and W. Wolf (G.P. 441,444, 6.2.26).—The coke is placed in a container tapered at the lower end and closed by a spring shutter. The coke is compressed by a piston working in the container, the shutter opening easily under the pressure and allowing the coke to pass without damaging it. A. B. Manning.

Preparing a material for use as pulverised fuel. F. Seidenschnur (E.P. 276,455, 8.6.26).—In order to produce a pulverised fuel with a high efficiency of combustion without having recourse to very fine grinding operations, the bituminous constituents of coal, lignite, etc. are removed by treatment with hot gases free from oxygen by processes previously described (cf. B., 1921, 803 a; 1925, 700), leaving a coke rich in volatile combustible constituents which need only be comparatively coarsely ground. C. O. Harvey.

Liquid fuels. A. Laurent (F.P. 615,749—50, 7.5.26. Belg., (a) 21.11.25, (b) 27.11.25).—Small quantities of (a) unsaturated cyclic hydrocarbons, *e.g.*, turpentine, cumene, eugenol, etc., or (b) ozone, or ozonides, which may be in solution in liquid hydrocarbons, are added to the fuel (mineral oils, benzene, alcohol, etc.) before or during combustion, producing thereby a considerable increase in calorific value. A. B. Manning.

Motor fuel. J. D. Riedel A.-G. (G.P. 441,455, 27.5.25).—The mixture of alcohols produced by the hydration, in a known manner, of the hydrocarbons formed by cracking acid resins and pitch residues from the distillation of aliphatic substances is used as a motor fuel, either alone or admixed with other fuels. A. B. Manning.

Red-coloured motor fuel. L. J. P. Guiaud (F.P. 617,225, 9.6.26).—A solution of 7·7 g. of naphthalene in 92·3 g. of benzene is treated with alkanna root and filtered. The filtrate is added to ordinary motor fuels in the proportion of 30 c.c. to 50 litres. A. B. Manning.

Composite motor spirit. Petroleum Chemical Corp., Assees. of C. K. Reiman (E.P. 253,131, 3.6.26. U.S., 4.6.25).—A motor fuel of high anti-knock properties is composed of a mixture of commercial gasoline, with 15—50% by vol. of the synthetic naphtha resulting from fractionation treatment of the product of cracking a petroleum distillate in the vapour phase, described in E.P. 273,781 (cf. B., 1927, 645). The naphtha is refined to polymerise the most reactive unsaturated hydrocarbons only, and usually contains less than 10% of aromatic hydrocarbons, with polymerides of amylene, butylene, and hexylene, boiling below 225°. R. C. Odams.

Manufacture of carbon black. C. A. Barbour, jun. (U.S.P. 1,643,736, 27.9.27. Appl., 1.3.26).—Hydrocarbons are burnt in a retort under a partial vacuum with sufficient air to form a substantial amount of free carbon, which is separated and collected while in the retort; the waste gases are then withdrawn. W. G. Carey.

[Producer gas] furnaces. H. J. Toogood, and R. Dempster & Sons, Ltd. (E.P. 276,410, 17.5.26).—A gas producer which is supplied (as usual) with air and steam over substantially the whole area of the grate is supplied with streams of returned waste gases round the edge of the grate, to form a protective gas layer along the walls. The waste gas may be returned by a fan. B. M. Venables.

Purification of gases from producers. L. A. E. Brodeur (F.P. 617,361, 10.6.26).—The gases are passed through a liquid and afterwards thoroughly cooled, the water vapour which then condenses serving to maintain the filter mass in a moist condition. A. B. Manning.

Separation of tar from gases. Nolze G.m.b.H. Gasreinigung u. Kühlerbau, Kaiserslautern (G.P. 441,493, 8.3.24).—The gas is cooled by tar directly after having been washed by the same tar warmed at least to the dew-point of the gas. A. B. Manning.

Apparatus for carbonising and distilling lignite, coal, bituminous shale, etc. Charbonnages et Agglomérés du Bassin de la Tave (F.P. 616,401, 20.3.26).—The material is charged into a retort through a double hopper, volatile products are withdrawn through tubes centrally disposed in each of the distillation tubes, and residues are removed by means of a screw conveyor. L. A. Coles.

Mineral oil distillation. Red River Refining Co., Inc., Assees. of J. E. Schulze (E.P. 257,250, 7.8.26. U.S., 24.8.25).—The high-vacuum process for the distillation of mineral oils claimed in E.P. 195,090 (B., 1925, 701) is applied to the production of high-grade, narrow-cut lubricating oils, particularly from crudes of high sulphur content. The distillation may take place in the presence of a neutralising agent such as lime, and under a pressure not exceeding 25 mm. (preferably below 5 mm.) of mercury. The lubricating oil vapours are condensed at a temperature just below their liquefaction point, and the uncondensed impurities taken off through a separate conduit. R. C. Odams.

Process of obtaining light hydrocarbons from hydroxylated, carboxylated, and like oxygenated organic compounds. J.-M. F. D. Florentin, A. J. Kling, and C. Matignon (E.P. 263,082, 26.5.26. Fr., 17.12.25).—Light saturated hydrocarbons are obtained by treating oxygenated compounds (*e.g.*, saponifiable oils, Autun shale oil) in the continuous liquid phase and at temperatures of 350—480° with hydrogen under pressures of 45 kg./cm.^{2} and over, in the presence of thoria as a catalyst. C. O. Harvey.

Process and apparatus for the cracking of oils. S. Seelig (E.P. 269,499, 11.3.27. Ger., 16.4.26. Addn. to E.P. 268,323; B., 1927, 805).—The original process is modified in that the oil is led through the helical tube and used as a cooling medium before entering the distillation apparatus. The helical tube is provided with an external baffle plate to increase its efficiency. R. C. Odams.

Method and apparatus for treating [cracking] hydrocarbons. C. P. Tolman (U.S.P. 1,643,036, 20.9.27. Appl., 26.8.26).—Hydrocarbon compounds are vaporised without substantial cracking taking place, and the vapours cracked by mixing with superheated mercury vapour. R. C. Odams.

Purification of hydrocarbons obtained by cracking processes. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 276,427, 26.5.26).—Benzines which remain water-white and odourless and which do not resinify are produced without loss of valuable aromatic and unsaturated hydrocarbons by refluxing cracked distillates for 30 min. with 2% of ferric chloride containing water (*e.g.*, the product obtained by melting the crystalline chloride). C. O. Harvey.

Treatment of gases produced in cracking hydrocarbons. Soc. Anon. d'Explosifs et de Prod. Chim., Assees. of L. Blanchet (E.P. 251,652, 30.4.26. Fr., 1.5.25).—The gases pass through successive operations of compression, cooling, etc. as follows:—(*a*) Cooling to about 5° under pressures of 2—5 kg. to effect condensation of the pentanes and amylenes; (*b*) condensation of the butanes and butylenes by cooling to about 0° under pressures of 10—20 kg.; (*c*) further cooling or absorption of propylene and ethylene by sulphuric acid or by hypochlorous acid to form the corresponding monochlorohydrins; and (*d*) chlorination of the residual gases to produce monochlorinated derivatives. The separated amylenes, pentanes, butylenes, and butanes are treated with hydrochloric acid and with chlorine to produce as far as possible only the monochlorinated derivatives, which may be converted by treatment with sodium acetate or by hydrolysis respectively into acetates or alcohols. C. O. Harvey.

Obtaining light hydrocarbons from animal or vegetable oils by the simultaneous action of heat, hydrogen under pressure, and a dehydrating catalyst. J.-M. F. D. Florentin, A. J. Kling, and C. Matignon (E.P. 276,007, 26.5.26. Fr., 17.12.25).—Light hydrocarbons are obtained from animal or vegetable oils by treating the oil in the continuous liquid phase at 350—480° with hydrogen at a pressure of at least 45 kg./cm.^{2} in the presence of a dehydrating catalyst, *e.g.*, thoria, alumina, clay, or silica. R. C. Odams.

Separation of paraffin wax from oils containing it. N. V. Bataafsche Petroleum Maatschappij, and J. F. P. Schönfeld (Dutch P. 14,653, 24.3.24. Conv., 16.1.25).—The oil and water are sprayed through separate atomisers so arranged that the sprays intermix, the temperature of the oil spray being about 3—10° higher than the setting point of the oil. L. A. Coles.

Manufacture of a medium for treating oils. H. Schlosstein (U.S.P. 1,638,643—4, 9.8.27. Appl., [A] 25.2.27; [B] 21.4.27).—(A) Bentonite clay mixed with magnesium chloride solution is treated with chlorine and sodium hydroxide, and the product, which contains aluminium and magnesium hypochlorites, is dried at the lowest possible temperature. (B) Bentonite clay mixed with magnesium hydroxide and calcium chloride solution is treated with chlorine. T. S. Wheeler.

Continuous treatment of minerals containing bitumen or petroleum. R. Amblard (F.P. 616,533, 14.10.25).—The material is charged continuously into a retort heated at such a temperature that a portion of the combustible content burns in a counter-current of air circulating through the retort. L. A. Coles.

Preparation of bituminous emulsions. H. E. Potts. From Mineral A.-G. Brig (E.P. 276,543, 3.12.26).—Bituminous emulsions suitable for road-making etc. are prepared by pouring molten bitumen etc. into a hot aqueous emulsion obtained by heating a glyceride (*e.g.*, bone fat) with excess of an aqueous alkali carbonate solution (preferably potassium carbonate). C. O. Harvey.

Plastic [bituminous] composition. C. S. Reeve, Assr. to Barrett Co. (U.S.P. 1,643,520—1, 27.9.27. Appl., 24.1.22).—(A) A mixture of coal-tar pitch and oil shale is heated at 340—350° for 5—10 hrs. (B) Oil shale is heated with coal-tar oil at 310—320° for 10—15 hrs. W. G. Carey.

[Treatment of fuel] alcohol, alcohol-containing mixtures, and similar liquids. W. Ostwald, Assr. to Benzol-Verband G.m.b.H. (U.S.P. 1,644,267, 4.10.27. Appl., 10.12.25. Ger., 4.9.25).—See E.P. 257,881; B., 1927, 245.

Manufacture of air gas from inflammable volatile liquids. H. Foersterling (E.P. 277,206, 5.3.26).—See U.S.P. 1,628,135; B., 1927, 625.

Method of distilling solid fuel. F. E. Hobson (E.P. 277,214—5, 10.1.27).—See U.S.P. 1,614,028; B., 1927, 290.

Bituminous emulsion. J. A. Montgomerie (U.S.P. 1,643,675, 27.9.27. Appl., 13.6.24. U.K., 8.12.23).—See E.P. 226,032; B., 1925, 91.

Resolution of water-in-oil emulsions. E. E. Ayres, jun., Assr. to Sharples Specialty Co. (U.S.P. 1,646,339, 18.10.27. Appl., 2.4.21).—See E.P. 195,876; B., 1923, 541 A.

Process of refining mineral oils. T. Hellthaler, Assr. to H. S. Riebeck Montan u. Ölwerke A.-G. (U.S.P. 1,643,272, 20.9.27. Appl., 1.8.25. Ger., 28.1.25).—See G.P. 426,157; B., 1926, 574.

Doors for closing retorts etc. Low-Temperature Carbonisation, Ltd., and J. F. Parker (E.P. 277,078, 7.6.26).

[Vapour outlet pipe and seal in] retorts for the distillation of coal and solid carbonaceous substances. Means for discharging retorts etc. LOW-TEMPERATURE CARBONISATION, LTD., and C. H. PARKER (E.P. 277,079 and 277,080, 7.6.26).

By-products from ammoniacal liquors (E.P. 276,393).—See III.

III.—TAR AND TAR PRODUCTS.

Benzol poisoning as an industrial hazard. L. GREENBURG (U.S. Public Health Rep., 1926, Reprint No. 1906. 63 pp.).

Naphthalene in gas. BONTE. **Lubricating oil from coal.** NIELSEN and BAKER.—See II.

PATENTS.

Recovery of pyridine, phenols, oils, and other by-products from gas liquor and like ammoniacal liquors. S. M. SHADBOLT, and CHEMICAL ENGINEERING & WILTON'S PATENT FURNACE CO., LTD. (E.P. 276,393, 1.12.26).—Gas and similar liquors are rendered harmless and fit for discharge into rivers etc. and valuable by-products are obtained by a process involving the addition of solvent oils such as benzine, benzene, paraffin, naphtha, creosote, etc. to the liquor prior to or during its distillation. The solvent oils are also added to the saturator in which the ammoniacal vapours are absorbed, and the vapours leaving the saturator also undergo treatment.
C. O. HARVEY.

Bituminous compositions (U.S.P. 1,643,520—1).—See II.

Treatment of wood (G.P. 438,944).—See IX.

IV.—DYESTUFFS AND INTERMEDIATES.

Oxidation of organic dyestuffs and of cellulose on exposure to light. V. SCHARVIN and A. PAKSCHWER (Z. angew. Chem., 1927, **40**, 1008—1010).—Pieces of porous porcelain dipped in solutions of various dyestuffs, *e.g.*, Methylene Blue, Crystal Violet, Congo Red, and Eosin, were sealed in an atmosphere of oxygen, nitrous oxide, or nitric oxide and exposed to the action of sunlight for four months. Considerable bleaching action had occurred in most cases, and in all cases carbon dioxide was found in the tube. Similar tests with cellulose (cotton wool) in oxygen showed the presence of carbon dioxide and oxycellulose in the tube after exposure for 30 hrs. to the light from a mercury vapour lamp. A. R. POWELL.

Detecting colours on wool. HIRST and KING.—See VI.

Fog formation by dyestuffs. LÜPPO-CRAMER.—See XXI.

PATENTS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G. Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 256,272, 3.8.26. Ger., 1.8.25).—2 : 3-Hydroxynaphthoic amide or arylamides (other than those derived from diaminodiaryls; cf. E.P. 218,568; B., 1924, 824) are coupled in substance or on the fibre with diazotised esters of 4-nitroanthranilic acid; *e.g.*, Naphthol AS with the methyl ester gives a bluish-red pigment, suitable for paper. C. HOLLINS.

Manufacture of finely-divided azo colouring matter [dyes] or lakes thereof. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 277,275, 12.4.26).—Azo dyes in finely-divided condition are obtained when the coupling is conducted in presence of a sulphonated alkylated aromatic hydrocarbon (*e.g.*, *iso*propylnaphthalenesulphonic acid) with or without the further addition of Turkey-red oil or other sulphonated oil. C. HOLLINS.

Manufacture of finely-divided solid materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 277,048, 12.4.26).—Finely-divided solid mineral or synthetic materials (excluding indigoid vat dyes) are obtained by dry grinding in the presence of aromatic sulphonic acids containing hydrocarbon side-chains or alkylated amino-groups, *e.g.*, *iso*propyl- and butylnaphthalenesulphonic acids, sulphonated phenol- or naphthalene-formaldehyde condensation products, dimethylmetanilic acid, diamylnaphthylaminesulphonic acid, etc. Turkey-red oil etc. may also be added. The process is especially suitable for lake printing colours.
C. HOLLINS.

Manufacture of benzanthrone derivatives. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, A. SHEPHERDSON, and S. THORNLEY (E.P. 276,766, 19.6.25).—Benzanthronesulphonic acid, obtained by direct sulphonation of benzanthrone with, *e.g.*, 100% sulphuric acid or oleum, in presence or absence of catalysts, such as mercury, boric acid, etc., is fused with alkali to give a reddish-blue vat dye. C. HOLLINS.

New black and grey vat dyestuffs [of the dibenzanthrone series]. BRIT. DYESTUFFS CORP., LTD., and S. THORNLEY (E.P. 276,767, 19.6.26).—The products of alkaline fusion of benzanthronesulphonic acid obtained according to E.P. 276,766 (preceding abstract) are condensed with hydroxylamine in concentrated sulphuric acid in presence or absence of ferrous sulphate, giving bluish-grey to black vat dyes. C. HOLLINS.

Manufacture of new vat dyes [of the dibenzanthrone series]. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, A. SHEPHERDSON, and S. THORNLEY (E.P. 276,768, 21.6.26).—The vat dyes of E.P. 276,766 (see above) are alkylated with or without a previous oxidation, or oxidation and reduction, to form vat dyes with deeper shade and superior fastness. Direct methylation gives a blue; oxidation and reduction give a green, loose to alkali, which is converted by methylation into a fast bluish-green. C. HOLLINS.

Process for preparing benzanthrone derivatives [dye for wool and acetate silk]. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 254,294, 15.6.26. Ger., 27.6.25).—3-Nitrobenzanthrone when boiled with sodium bisulphite solution is converted into a sulphonic acid of 3-aminobenzanthrone. The product dyes wool, silk, and acetate silk in bright red shades. C. HOLLINS.

Production of grey to black vat dyes [of the dibenzanthrone series]. L. B. HOLLIDAY & CO., LTD., and C. SHAW (E.P. 277,125, 15.6.26).—Grey to black vat dyes are obtained by chromic acid oxidation of dibenzanthrone or of the oxidation products obtained by previous treatment with nitric and sulphuric acids, or with manganese dioxide and sulphuric acid. C. HOLLINS.

Manufacture of [thioindigoid] vat dyes. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 260,940, 25.5.26. Ger., 7.11.25. Addn. to E.P. 251,996; B., 1927, 809).—The thioindigoid vat dyes of the earlier patent are halogenated, *e.g.*, by chlorosulphonic acid, whereby the shade becomes bluer and the fastness to kier-boiling is increased.

C. Hollins.

Manufacture of anthraquinone derivatives. I. G. Farbenind. A.-G., R. E. Schmidt, and R. Berliner (E.P. 277,109 and 277,110, 10.6.26. Addn. [A] to E.P. 244,462 and [B] to E.P. 244,463; B., 1927, 674).—(A) The two series of products from 4-substituted 1-aminoanthraquinones and formaldehyde in acid media, described in the prior patent, are obtained more readily by using sulphuric acid in presence of a metallic reducing agent (copper, zinc, aluminium), the change from the known, unstable primary products to the new, stable products being thereby facilitated. (B) For the oxidation, nitric or nitrous acid is preferred to the oxidants mentioned in the prior patent. C. Hollins.

Preparation of 2-chloroquinizarin. Newport Co. (E.P. 260,544, 1.5.26. U.S., 2.11.25).—3 : 4-Dichlorophenol is condensed with phthalic anhydride in presence of sulphuric and boric acids at about 200°, and the resulting boric ester is hydrolysed to give 2-chloroquinizarin, m.p. 235—236°. C. Hollins.

Manufacture of dyes [related to indulines and nigrosines]. H. T. Bucherer (E.P. 252,745, 31.5.26. Ger., 29.5.25).—Black dyes are obtained by heating an aromatic nitro-compound with an arylamine in sulphuric acid in the proportion of 1 nitro-group to 2 amino-groups; *e.g.*, nitrobenzene with aniline or α- or β-aminoanthraquinone; nitrobenzene with 1 : 5- or 2 : 6-diaminoanthraquinone (1 mol.); dinitroanthraquinone with aniline (4 mols.), α- or β-aminoanthraquinone (4 mols.), or diaminoanthraquinone (2 mols.).

C. Hollins.

Naphthylaminoalkylamines. W. Duisberg, W. Hentrich, L. Zeh, and J. Huismann, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,633,929, 28.6.27. Appl., 17.4.25. Ger., 21.5.24).—See E.P. 249,717; B., 1926, 433.

Catalytic reduction of organic nitro-compounds. M. Kahn and R. Mayer, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,639,186, 16.8.27. Appl., 14.8.26. Ger., 29.9.24).—See E.P. 260,186; B., 1927, 8.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Influence of plasticising on the mechanical-elastic properties of artificial and natural plastic substances. III. Cellulose materials. O. Manfred and J. Obrist (Kolloid-Z., 1927, 43, 41—46. Cf. A., 1927, 514; B., 1927, 563).—Investigation of a number of cellulose materials, including various samples of celluloid and of cellulose acetate (cellon, rhodoid, etc.) confirms the results previously obtained with artificial horn and synthetic resins (*loc. cit.*). It is found that the elastic properties of plastic substances are, apart from factors such as the temperature, the nature of the starting material, etc., functions of the work of plasticising, *i.e.*, the total mechanical and chemical work required to bring about the changes of disaggregation and re-aggregation leading to the formation of the plastic mass. With decreasing work of plasticising, the elasticity modulus rises. The technical methods for bringing cellulose or its ester into a plastic state are discussed from the point of view of the mechanical and chemical disaggregation and re-aggregation, with special reference to the action of plasticising agents. L. L. Bircumshaw.

Determination of wood gum in celluloses. H. Bubeck (Papier-Fabr., 1927, 25, 617—620).—Reliable results for the determination of wood gum (*i.e.*, the portion soluble in 5% sodium hydroxide and precipitated on neutralisation of the extract with acid) are only obtained if the cellulose is reduced to a fine state of division and a constant temperature is maintained during the extraction. If the extract is neutralised and precipitation is carried out with alcohol, the latter must not be diluted with water, since, for example, higher values are given for precipitation with 96% than with 88% alcohol. Provided the cellulose is in the form of wadding, and the temperature is constant at 15°, extraction is completed in 1—2 hrs. instead of 2—3 days, whilst the laborious operations of collecting and weighing the precipitate are avoided by the use of an oxidation method for the determination of the dissolved material. In the procedure recommended, 5 g. of cellulose are treated with 100 c.c. of 5 vol.-% sodium hydroxide at 18° for 1—2 hrs., the mixture is filtered, 25 c.c. are withdrawn from the filtrate, oxidised with excess of 1·5*N*-chromic acid containing sulphuric acid, and an aliquot part of this reaction mixture, after suitable dilution, is titrated by the usual iodometric method. 1 c.c. of the chromic acid solution corresponds to 10·13 mg. of wood gum. The differences between the results given by this method and by the neutral (precipitation) method are characteristic of the degree of bleaching of the material, unbleached cellulose showing the highest, highly bleached the lowest, and weakly bleached intermediate values.

B. P. Ridge.

Stretching processes for [viscose] artificial silk. Duré (Kunstseide, 1927, 9, 459—460).—The dyeing and physical properties of wet, freshly coagulated viscose silk may be advantageously modified by stretching previously to the usual processes of washing, desulphurisation, bleaching, and drying. Wet viscose silk can be stretched 1·5 times in length without breakage at a rate of less than 50 m./min., but preferably at 40 m./min., by winding from one drum to another revolving at a higher speed. Stretched, wet, freshly coagulated viscose silk suffers a shrinkage during the subsequent processes of purification and drying which diminishes with increase of stretching; it also has a decreased elasticity, but the decrease is negligible when the stretching is less than 1·4 times. Stretched silk has an increased tensile strength, particularly when the stretching is within the limits 1·25—1·40 times. The most satisfactory results are obtained by stretching 1·4 times.

A. J. Hall.

Nature of paper formation. G. Porrvik (Papier-Fabr., 1927, 25, 589—596).—The splitting up of wood during grinding takes place in three different ways, viz., (1) the brushing out of the fibres lengthwise, (2) trans-

verse breaking up into short pieces, and (3) wearing away with the formation of mucilage. With ordinary pressure in the hollander the first type predominates, with high pressure the second, with no pressure the third, whilst the ball mill gives a mixture of these. By suitably altering the degree of grinding, as high values for strength etc. can be obtained for a highly bleached sulphite cellulose as for a strong-fibred material. The disadvantage of an overcooked or overbleached product lies not so much in the weakness of the fibres as in the fact that it reaches the required degree of grinding too easily and swells and forms clods too readily. The final degree of grinding depends partly on the original and partly on the acquired composition of the material. Up to 30% loss of strength may result through drying on the machine, air-drying being less harmful, whilst the permissible temperature depends upon the nature of the material. This loss of strength is considered to be due to the fact that the material is composed of fibres and mucilage, the drying hardening the latter to an almost irreversible form. This hardening is overcome by subsequent bleaching, an increase of 8% in strength and a decrease of 10% in the necessary time of grinding being recorded for a treated sample. The addition of sodium hydroxide during grinding shortens the time required, but decreases the strength (compared with that when no addition is made); if the time of treatment is prolonged, however, the strength of the material increases. Inferior results are given for the addition of hydrochloric acid. The increase in the strength of paper produced by a suitably long, as opposed to a short, time of grinding is attributed to the fact that, although the fibres are shortened, the felting capacity of the material is increased, Strength also depends upon the flexibility of the fibres. In a series of samples, the strongest paper was found to have the shortest fibre lengths but the greatest flexibility of fibre. B. P. Ridge.

Lignocell paper. von Possanner (Papier-Fabr., 1927, **25**, 601—603).—Lignocell paper may be produced by only slight modification of the apparatus used for normal steamed mechanical pulp. Its natural colour is very light, and it may therefore be dyed light shades and used for many purposes for which the dark-coloured, steamed, mechanical pulp is unsuitable. Lignocell does not behave like cellulose, but as a true wood pulp, and shows all the characteristic properties and staining tests of the latter. For the working-up of the half-stuff no severe hollandering is necessary as the material has only to be opened and mixed; beating is therefore essential. In spite of its agglutinous nature it behaves well on the paper machines, and owing to its good fibre felting qualities greater working velocities are possible than in the production of ordinary wood cellulose papers. Results of tests show the strength of lignocell paper to lie between those of ordinary log-wood paper and cellulose packing paper, whilst as its volume/weight ratio is large it is specially suitable for the production of voluminous papers. B. P. Ridge.

Oxidation of cellulose. Scharwin and Pakschwer. —See IV.

Cellulose in soil. Waksman.—See XVI.

Patents.

Process for treating green or dry stalks of ramie and other vegetable fibres. Soc. Civile des Proc. Masse (E.P. 266,344, 15.2.27. Fr., 22.2.26).—The fibres are boiled in alkali lye (d 1·04) in an autoclave, passed through a soap bath, dried, and are then treated repeatedly in a bath of rice starch, potato flour, or the starch of other amylaceous plants, again dried, and beaten lightly to detach the excess of starch. W. G. Carey.

Manufacture of pulp from fibrous material. F. K. Fish, jun. (U.S.P. 1,632,467, 14.6.27. Appl., 15.7.25; cf. E.P. 255,030; B., 1927, 474).—A sulphite wood-pulp digester is provided with an auxiliary tank into which the pressure is suddenly relieved at intervals, thus promoting disintegration of material. T. S. Wheeler.

Fabric cement. N. C. Amen, Assr. to H. H. Randolph (U.S.P. 1,643,437, 27.9.27. Appl., 10.7.24).—See E.P. 258,698; B., 1927, 9.

Extracting pure cellulose from bagasse of sugar cane. E. C. H. Valet (E.P. 277,163, 25.8.26).—See U.S.P. 1,630,147; B., 1927, 552.

Apparatus for conditioning textile fibres. W. W. Groves. From Borne Scrymser Co. (E.P. 277,213, 5.1.27).

Ring spinning device for spinning continuously-fed textile fibres. G. B. Ellis. From Soc. pour la Fabr. de la Soie "Rhodiaseta" (E.P. 277,151, 29.7.26).

Drying, calendering, and like machines. S. Milne (E.P. 277,181, 9.10.26).

Solubilising carbohydrate ethers (E.P. 277,111). —See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Fastness to light of dyestuffs on woollen and worsted fabrics. V. Effect of ultra-violet radiation on the fading of dyed fabrics. L. Hill. **VI. Transmission of sunlight through glass and its effect on fading of dyestuffs.** I. O. Griffith and R. G. C. Jenkins (J. Soc. Dyers and Col., 1927, **43**, 296—302, 297—299).—V. The amount of fading produced by exposing worsted fabrics dyed with Indigo Carmine X, Ponceau RG, and Neolan Green B to light from a mercury vapour lamp was chiefly dependent on the proportion of ultra-violet light emitted, the fading being greater for light of smaller wave-length. Vitaglass transmits most of the light in sunlight which produces fading. Fading occurs more rapidly in the presence of moisture.

VI. The transmissivities of light through English and foreign window plate glass such as would be used for shop windows displaying coloured textile materials are similar, 90—95% of the visible spectrum, a small proportion of the ultra-violet (3500—3000Å.), and scarcely any of the far ultra-violet (3000—2900Å.) being transmitted. Vitaglass transmits the entire spectrum with but a small loss of intensity, its transmissivities for light of 7000—3250Å. and 2900Å. being 90 and 72·5% respectively Polished quartz has transmissivities of 90 and 84% for light of 7000—2200Å. and

2000Å. respectively. Vitaglass is considered satisfactory for covering and protecting dyed fabrics during fading tests. A. J. Hall.

Effect of light on [indigo]-coloured cotton fabric. I. (Miss) E. Hibbert (J. Soc. Dyers and Col., 1927, **43**, 292—294).—Plain calico and similar fabrics dyed with Brilliant Copper Blue, Chlorazol Sky Blue, and Benzopurpurin 4B had nearly the same copper numbers (3·6—3·8) after exposure to 669 hrs. of bright sunshine. Since complete destruction of the dyes occurred long before the copper number of the fabric reached 3·6, it is concluded that the fading of dyed cotton is not due to the reducing action of the cellulose under the influence of light (cf. Harrison, B., 1914, 689). Certain dyes retard or accelerate the tendering of cotton under the influence of light. *E.g.*, cotton fabrics dyed with Duranthrene Yellow, Cibanone Yellow, and Indigo, and having an original tensile strength of 48 lb., had tensile strengths of 27, 16, and 22 lb., respectively, after exposure for 120 hrs. in a Fadcometer. Isatin was obtained in a pure crystalline state by extraction with water and ether of calico dyed with indigo and exposed to light. Chromium hydroxide in cotton materials dyed with Chrome Green had a marked protective effect against the action of sunlight. A. J. Hall.

Dyes not fast to ironing. R. Haller (Kolloid-Z., 1927, **43**, 47—50; cf. B., 1926, 402).—The theory previously advanced (*loc. cit.*), that the behaviour of dyes which are not fast when brought into contact with heated metal is due to the multidispersity of the dyes, has been further tested. It has been found possible to separate strong solutions of a number of dyes, including Congo Red A, Diamine Blue 3R, Azo Blue, Congo Corinth, Bordeaux Extra, etc., into two fractions by exhaustive dialysis. In each case a blue precipitate was obtained, dissolving fairly easily in water on addition of electrolytes, and a red filtrate. Samples of fabric were dyed in the respective filtrates and precipitate solutions, and the tints so obtained were compared by means of a Zeiss step-photometer. The behaviour of the dyed fabrics when brought into contact with heated metal was also investigated colorimetrically. The results are somewhat complex, but support the view that the phase of low degree of dispersion has a strong tendency to pass over into that of higher degree of dispersion.

L. L. Bircumshaw.

Stretching artificial silk. Duré.—See V.

Photographic images on cellulose acetate film. Hall and Hill.—See XXI.

Patents.

Treatment of [immunised] cotton and viscose silk fibres preparatory to dyeing. P. Karrer (E.P. 263,169, 15.12.26. Addn. to E.P. 249,842; B., 1927, 71).—The preparation of amidated cotton and viscose silk having an affinity for acid dyes, as described in the chief patent, is modified so that instead of treating the esterified (immunised) fibres for a short time at high temperatures with solutions of the bases mentioned, the esterified fibres in their wet state are directly treated with ammonia gas or vapours of suitable amines. Amidation may also be effected with pyridine, piperidine, quinoline, or other heterocyclic base. *E.g.*, cotton partially esterified with benzenesulphonic acid groups is exposed to ammonia gas at 90°, whereby it acquires a marked affinity for acid dyes (cf. Karrer and Wehrli, B., 1926, 659). A. J. Hall.

Dyeing of [immunised] cotton materials. Sandoz Chemical Co., Ltd., and A. E. Woodhead (E.P. 274,276, 19.7.26. Addn. to E.P. 246,609; B., 1926, 270).—The dyes and methods of dyeing described in the chief patent are also suitable for dyeing cotton immunised by the methods described in G.P. 346,883, E.P. 195,619, 224,502, and 241,854 (B., 1922, 214 A; 1924, 128; 1925, 352; 1926, 270). A. J. Hall.

Vat dyeing. J. W. Leitch & Co., Ltd., A. E. Everest, and J. A. Wallwork (E.P. 274,550, 19.4.26).—Textile materials containing fibres such as wool and cellulose acetate silk, which are deleteriously affected by the caustic alkalis usually present in vat dye liquors, are dyed with vat dyes from dye solutions prepared with a hyposulphite, an alkali salt of a fatty acid having a high mol. wt., *e.g.*, stearic acid, and ammonia or soda ash. The method is suitable for dyeing with anthraquinone, and especially indigoid vat dyes, and since no caustic alkali is present the dyeing may be effected at comparatively high temperatures, *e.g.*, 50—60°. A satisfactory dye liquor for dyeing indigo is prepared at 70—80° with 100 g. of stearine soap, 10 litres of water, 100 g. of 20% indigo paste, 500 c.c. of ammonia solution (*d* 0·88), and 50 g. of sodium hyposulphite. Two-colour effects are obtained by using vat dyes having affinities for particular fibres. *E.g.*, Algol Black RO dyes cotton and viscose silk satisfactorily, but only stains wool and cellulose acetate silk; Leucole Yellow G and Algol Brilliant Violet R dye cotton, wool, and viscose silk, but only feebly stains cellulose acetate silk.

A. J. Hall.

Dyeing and printing of vat colours. J. S. Wilson, J. Thomas, and Scottish Dyes, Ltd. (E.P. 274,178, 12.4.26).—Two-coloured effects are obtained by suitably discharging fabrics dyed with mixtures of vat dyes containing those usually dyed from a hot, strongly alkaline dye liquor, and those dyed from a cold, weakly alkaline liquor, the former class of vat dye being much less readily discharged than the latter by means of Leucotrope and reducing agents such as sodium formaldehyde-sulphoxylate. In preparing the ground shade before discharging, the two classes of dyes are dyed together regardless of their usually different methods of application. *E.g.*, yellow effects on an orange ground are obtained by dyeing cotton fabric with a mixture of Caledon Yellow G and Caledon Red FF at 35°, then printing with a discharge paste containing 23 pts. of 25% British gum thickening, 4 pts. of zinc oxide, 5 pts. of Leucotrope W, 12 pts. of sodium formaldehyde-sulphoxylate, 1 pt. of anthraquinone, and 5 pts. of caustic soda, drying, steaming, and then passing through a boiling 0·1% solution of caustic soda. Suitable hot-dyeing strong caustic vat dyes include Caledon Blue R, Caledon Brilliant RR, Caledon Jade Green, and Caledon Gold-Orange G, and cold-dyeing weak caustic colours include Caledon Red FF, Caledon Blue 3 RK, Caledon Olive R, Caledon Yellow 3 G, and Caledon Brilliant Violet R.

A. J. Hall.

Dyeing artificial [regenerated cellulose] silk. Brit. Dyestuffs Corp., Ltd., J. Baddiley, P. Chorley, and C. Butler (E.P. 276,757, 7.6.26. Addn. to E.P. 276,450; B., 1927, 812).—Level shades on viscose silks are obtained by using secondary disazo dyes of the type: aminosalicylic acids or homologues → usual middle components → sulphonated naphthols, naphthylamines, or *N*-derivatives of the latter. The shades are blue to violet, brown, and black. C. Hollins.

Wetting-out agents or emulsifiers, and their use in the textile and other industries. Brit. Dyestuffs Corp., Ltd., J. Baddiley, and E. Chapman (E.P. 274,611, 3.6.26).—Wetting-out agents are prepared by sulphonation of the condensation products of a raw or purified mineral oil (*e.g.*, certain fractions of such oils obtained during purification or cracking) with a suitable aliphatic, alicyclic, or aralkyl alcohol; alternatively, the mineral oil may be sulphonated before condensation. The sulphonated products may be left in the form of the free acid, or converted into sodium salts by liming-out in the usual manner. The products show strong wetting-out properties even at a dilution below 0·5%. Various applications of these processes are shown by 12 examples. A. J. Hall.

Apparatus for dyeing felt or other hats. G. Charles (E.P. 276,893, 4.3.27).

Aralkylated ethers (E.P. 277,098).—See XX.

Decoration of fabrics (G.P. 441,150).—See XXI.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Determination of sulphide, thiosulphate, and sulphur in insoluble carbonates, especially in commercial barium carbonate. H. Brintzinger and F. Rodis (Z. anal. Chem., 1927, **71**, 434—441).—Barium carbonate, obtained by saturating with carbon dioxide the aqueous extract of the mass produced by heating barytes with coal dust, almost invariably contains adsorbed barium sulphide together with its oxidation products, barium thiosulphate and free sulphur. For the determination of sulphide 10 g. are dissolved in 100 c.c. of 2*N*-sulphuric acid and the evolved hydrogen sulphide is collected in cadmium acetate–acetic acid solution and determined iodometrically as usual. The insoluble residue consists of barium sulphate containing the free sulphur originally present and that produced by the decomposition of the thiosulphate; the sulphur is determined by digesting the mass for 20 min. on the water-bath with 50 c.c. of 0·01*N*-permanganate, which oxidises it to sulphuric acid, and titrating the excess permanganate with oxalic acid. The sum of the thiosulphate and sulphide present is obtained by treating 10 g. of the carbonate with 50 c.c. of 0·01*N*-iodine and sufficient hydrochloric acid to dissolve the barium compounds, and determining the excess of iodine with thiosulphate. From the data obtained the composition of the material is readily calculated if the total soluble barium is determined acidimetrically as usual. A. R. Powell.

Manufacture of ferric aluminium [sulphate] by the Argentine Sanitary Works and Water Supply. M. L. Negri and A. A. Bado (J. Amer. Water Works' Assoc., 1927, **18**, 287—299).—Ferric aluminium sulphate for use as coagulant at the Recolcta Plant is manufactured from the Pampeano loess which is found in large quantities under the vegetal cover in the province of Buenos Aires. The loess, which is essentially a double silicate of iron and aluminium, contains about 17% Al_2O_3 and 7% Fe_2O_3. Sulphuric acid (70% H_2SO_4) is heated by means of steam to 100°, and the mineral is gradually added, the mixture meanwhile being well stirred by compressed air. When the mass is homogeneous, the stirring is stopped and the heating continued until 110° is reached. Steam injection is then discontinued, and the mass is kept for 20 hrs. The extraction of the ferric aluminium sulphate is subsequently effected by separate washings with cold water, settlement, and decantation. After further settlement to deposit fine clay in suspension, the liquid is used direct for water treatment or concentrated to *d* 1·84. The compact green mass which solidifies from the concentrated liquor on cooling contains about 17% of mixed oxides. W. T. Lockett.

Ammonia and sulphur from gas. Burkheiser.—See II.

Patents.

Production of complex hydrofluoric acids. A. F. Meyerhofer (E.P. 250,211, 19.3.26. Ger., 1.4.25).—Complex fluorides are decomposed without agitation by heat, under pressure, in the presence of catalysts and of hydrogen or gaseous hydrogen compounds, excluding solid carbonaceous fuels. A mixture which evolves hydrogen or gaseous hydrogen compounds and which yields by-products may be used, *e.g.*, ammonia to form nitrides, steam to form oxides, volatile acids to form salts; further, the use of a gas, such as furnace gas, having reducing properties, or of certain oxidising mixtures such as air, or of various other gaseous mixtures, is claimed. Salts, *e.g.*, alkali chlorides, may be added to the fluorides to combine the hydrofluoric acid with the alkali and to obtain as a by-product an acid which does not contain fluorine. W. G. Carey.

Concentration of sludge acid. A. A. Vineyard (U.S.P. 1,636,724, 26.7.27. Appl., 27.11.25; cf. U.S.P. 1,596,907; B., 1926, 914).—The method described in the patent cited is applied to the concentration of sludge acid. T. S. Wheeler.

Manufacture of alkaline potassium compounds. W. Siegel (U.S.P. 1,636,710, 26.7.27. Appl., 21.10.25. Ger., 12.7.24).—Potassium fluosilicate is treated with potassium hydroxide solution, *d* 1·125, and the silica separating is collected on a filter. The filtrate is treated with calcium oxide to yield a solution of potassium hydroxide, a part of which is returned to the process, and a precipitate of calcium fluoride, which is heated with dilute sulphuric acid and silica under pressure to give a solution of hydrofluosilicic acid. This is treated with potassium chloride solution to precipitate potassium fluosilicate. T. S. Wheeler.

Converting a cyanide compound [calcium cyanide] into ammonia. G. H. Buchanan and J. L. Osborne, Assrs. to Amer. Cyanamid Co. (U.S.P. 1,638,522, 9.8.27. Appl., 18.12.23).—Crude calcium cyanide is heated with water under a pressure of 4 atm.

Most of the ammonia produced is removed at this pressure, and the remainder is expelled from solution by treatment with steam. T. S. WHEELER.

Treatment of lithium-containing silicates by means of neutral alkali salts. METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 269,878, 31.3.27. Ger., 22.4.26).—Water-soluble lithium compounds are obtained from silicates containing lithium with the use of less alkali salt than heretofore if definite temperature limits of 700—950° are maintained in a mechanical roasting furnace, rotary drum furnace, or Dwight–Lloyd furnace. W. G. CAREY.

Manufacture of precipitated zeolites. H. KRIEGSHEIM and W. VAUGHAN, Assrs. to PERMUTIT Co. (U.S.P. 1,642,880, 20.9.27. Appl., 17.8.25).—A gel of normal structure, produced by the action of a solution of sodium aluminate on a solution of sodium silicate, is partly dried without preliminary separation of contained water, and is then washed and dried. H. HOLMES.

Manufacture of base-exchange silicates. P. N. ENGEL (E.P. 277,082, 7.6.26).—A non-alkaline salt of an amphoteric hydroxide, *e.g.*, aluminium sulphate, is treated with an acid, and a solution of an alkali silicate, *e.g.*, sodium silicate, is added. The proportion of acid is such that the molecular ratios in the product are one of alkali metal oxide and one of amphoteric metal oxide to 10 or more of silica, and the mixture is made alkaline to phenolphthalein by varying the amount of the amphoteric compound. The resulting gel is dried at below 100° (preferably 80°), the soluble compounds are dissolved out, and the dried gel is subjected to steam treatment to resolve it into particles. W. G. CAREY.

Manufacture of carbon dioxide. S. E. ELKIN, Assr. to P. E. HAYNES (Can. P. 264,697, 14.9.25).—Charcoal, with the addition of calcium carbonate to regulate the temperature, is burnt in a current of pure oxygen, the highly-compressed gases thus obtained are cooled to liquefy the carbon dioxide, and the remaining gas is allowed to expand and thus cool further quantities of the gas mixture. A. R. POWELL.

Centrifugal apparatus [for separation of gases]. SYNTHETIC AMMONIA & NITRATES, LTD., and H. A. HUMPHREY (E.P. 276,557, 15.1.27).—A centrifugal rotor which may be run at peripheral speeds of the order of 600 m./sec. comprises two conical discs fitting base to base on a common shaft, leaving a space between which is divided by partitions to ensure proper whirling of the material. The thickness of the metal increases towards the axis. The feed mixture is introduced at one end and the lighter constituent withdrawn at the other end of the hollow shaft. The heavier constituent is exhausted through passages in the rim so great that the outlets are substantially tangential, and the kinetic energy of the issuing product helps to drive the rotor. The centrifugal force developed is so great that a partial separation of permanent gases may be effected, but the apparatus is very efficient if the temperature is lowered below the critical temperature of one constituent, the pressure due to centrifugal force, combined if necessary with some initial pressure, being sufficient to liquefy that constituent. The latent heat evolved passes through the walls of the rotor to the issuing heavy product, which, after cooling by expansion through the outlet nozzles, is brought back in a comparatively stationary state over the outside of the rotor, between it and the fixed casing. This latent heat also serves to prevent freezing of, *e.g.*, carbon dioxide, which can be removed from air with an efficiency approaching 100%. B. M. VENABLES.

Manufacture of fluorides. F. SANDER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,642,896, 20.9.27. Appl., 8.6.25. Ger., 10.6.24).—See E.P. 257,981; B., 1926, 979.

Manufacture of ferric sulphate. B. HART (U.S.P. 1,644,250, 4.10.27. Appl., 18.10.26. U.K., 12.6.26).—See E.P. 273,883; B., 1927, 653.

Conversion of lead chloride into lead carbonate. S. C. SMITH (U.S.P. 1,643,261, 20.9.27. Appl., 14.5.24. U.K., 30.1.24).—See E.P. 272,053; B., 1927, 555.

Exothermic reactions (E.P. 268,722).—See I.

Copper from sulphate solution (U.S.P. 1,643,922).—See X.

Electrolysis of alkali chlorides (E.P. 267,560 and 269,513). **Electrolysis of water** (E.P. 254,268).—See XI.

VIII.—GLASS; CERAMICS.

Mechanical manufacture of window glass. M. BABILLE (Chim. et Ind., 1927, **18**, 371—380).

Colouring agents in glasses and glazes. (Sir) H. JACKSON (Proc. Roy. Inst., 1927, **25**, 230—241).

Corrosion of silica retorts. DIAMOND.—See II.

Fading of dyes (HILL, also GRIFFITH and JENKINS).—See VI.

PATENTS.

Production of ceramic masses. DEUTS. TON- & STEINZEUG-WERKE A.-G., Assees. of T. KÜRTEN (G.P. 441,944, 15.11.25).—The addition of powdered ferrosilicon to ordinary ceramic masses improves the mechanical properties and the resistance to temperature changes of fired articles made from the mixture. A. R. POWELL.

Refractory, acid-proof, and other ceramically bonded products. SCHEIDHAUER & GIESSING A.-G. (E.P. 264,192, 11.1.27. Ger., 11.1.26. Addn. to E.P. 263,194; B., 1927, 702).—A colloidal solution of substances of the same nature as the non-plastic material used to make the products is mixed with the latter as binding material, so that a non-castable mass is obtained which is moulded by beating, stamping, pressing, or other strong mechanical treatment to ensure close contact, after which the products are dried and burnt. W. G. CAREY.

Manufacture of glass or glass-like objects. E. HOPE, Assr. to F. W. ATACK (U.S.P. 1,644,131, 4.10.27. Appl., 26.12.25. U.K., 26.1.25).—See E.P. 254,668; B., 1926, 747.

IX.—BUILDING MATERIALS.

Diffusion of water-soluble substances in impregnated wood. R. NOWOTNY (Z. angew. Chem., 1927, **40**, 1060—1062).—Graphical representations of the results of experiments made by W. F. Sherefesee (Forest

Serv. Circ. 132, 1908) on woods soaked in zinc chloride solution show that, contrary to the conclusions of this author, there is a slow but distinct diffusion of zinc chloride after removal of the wood from the solution. When hemlock spruce-fir was steeped under pressure, the zinc chloride solution penetrated, in a longitudinal direction only, to a distance of 1 ft., but no further. In the case of American larch, penetration took place in gradually diminishing amounts to distances of 3 ft. and more. After keeping duplicate halves of the wood samples for periods of 3½ and 4½ months, sections within 1 ft. of the steeped end showed a lower zinc chloride content, whilst those further removed from this end were found to contain more zinc chloride than before, the salt tending in course of time to distribute itself uniformly through the wood. F. R. ENNOS.

Blast-furnace slag as ballast. H. BURCHARTZ and G. SAENGER (Stahl u. Eisen, 1927, **47**, 1663—1664).—The composition of slag from iron blast furnaces in Germany varies within the following percentage limits: 30·8—35·6% SiO_2, 9·1—12·0% Al_2O_3, 0·3—0·8% FeO, 1·3—3·5% MnO, 35·9—43·4% CaO, 3·7—10·2% MgO, 0·1—0·3% SO_3, 1·1—1·8% S, and 0—0·34% P_2O_5. It has *d* 3·0, apparent sp. gr. 1·14—2·95, and absorbs 1·1—2·8% of its weight of water. Its resistance to frost and to shattering during use as railway ballast is equal to that of granite, but slightly poorer than that of basalt. During the first year on the track it tends to accelerate the rusting of iron, but subsequently becomes inert. A. R. POWELL.

Physical chemistry and the problem of roads. G. BAUME (Bull. Soc. chim. Belg., 1927, **36**, 469—484).—A discussion of the surface treatment of roads.

Action of cement and gypsum on light metals. MEYER and PUKALL.—See X.

PATENTS.

Composite building and insulating materials. G. ARNESEN and P. A. BECH (E.P. 269,845, 19.11.26. Norw., 26.4.26).—Sawdust or wood chips, cement, calcium oxychloride, and active silica are mixed together in a dry condition, and water is subsequently added. W. G. CAREY.

Treatment of woods impregnated with tar oil. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of A. VAUPEL (G.P. 438,944, 11.10.25).—The tar oil with which the wood of telegraph poles etc. is impregnated is removed from a surface layer of 0·5—1 mm. depth by treatment with a suitable solvent or by steaming. This surface layer is then re-impregnated with a sodium silicate-magnesite mixture, collodion, or similar preparation. This prevents escape of tar oil from the interior and forms a weatherproof outer layer, which at the same time is harmless to workers handling the wood. A. B. MANNING.

Manufacture of hydraulic cement. H. S. SPACKMAN (U.S.P. 1,643,137, 20.9.27. Appl., 17.12.24).—See E.P. 244,756; B., 1927, 367.

Manufacture of cementitious material. H. S. SPACKMAN (U.S.P. 1,643,136, 20.9.27. Appl., 22.9.23).—See E.P. 222,151; B., 1925, 503.

Preparing a material suitable for use in the manufacture of moulded articles. S. J. HAYDE, Assr. to AMER. AGGREGATE CO. (Re-issue 16,750, 27.9.27, of U.S.P. 1,255,878, 12.2.18).—See B., 1918, 243 A.

Granulating molten [blast-furnace] slag [for cement]. F. SPIES (E.P. 277,154, 4.8.26).

Bituminous emulsions (E.P. 276,543).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Corrosion of iron in tap water. J. TILLMANS, P. HIRSCH, and W. WEINTRAUD (Gas- u. Wasserfach, 1927, **70**, 845—849, 877—884, 898—904, 919—925).—In the absence of oxygen the rate of corrosion of iron in water increases with decrease of p_H, but in the presence of oxygen the rate is independent of the p_H. As there is a copious deposition of a protective film of rust in alkaline water and no deposition in acid water, it follows that oxygen acts as an accelerator of corrosion in alkaline water. The first action of water on iron is accompanied by the formation of ferrous ions which retard further action; oxygen causes these to be converted into ferric hydroxide in alkaline solutions, and thus their retarding influence is eliminated. In air-free water the rate of corrosion of iron is governed by the rate of discharge of the hydrogen ions first formed, which is relatively slow; oxygen acts as a depolariser in this action. The reaction $O_2 + 2H_2O = 4OH' + 4(+)$ is subject to less retardation than that in which molecular hydrogen is evolved, and hence the influence of the latter action on the rate of corrosion is removed; this theory explains the absence of hydrogen evolution when iron corrodes in water containing oxygen. As, however, the oxygen ionisation reaction is dependent on the p_H, it cannot be the governing factor in the mechanism of corrosion; this is most likely to be the rate of diffusion of oxygen to the metal surface and the rate at which ferrous ions are formed, the former being independent of the p_H and the latter only indirectly dependent, being governed by the difference between the oxygen ionisation potential and the solution potential of the iron. With high concentrations of oxygen the metal becomes passive; the oxygen concentration necessary to produce this state is lower the higher the p_H, and lower in moving water than in still water. The presence of small quantities of electrolytes increases the rate of corrosion by assisting in flocculating the colloidal products of corrosion and by increasing the conductivity of the solution and so enhancing the action of local elements. Free carbon dioxide acts as an accelerator on the corrosion as it tends to prevent the formation of protective films; its action may be eliminated and the formation of a protective film accelerated by passing the water through a layer of marble chips. Aerated water so treated rapidly forms a tenacious film of ferric hydroxide and finely-crystallised calcium carbonate on the inner surface of an iron pipe, which is thereby efficiently protected from further attack. On this account soft waters should be neutralised with limestone or marble in preference to sodium carbonate or hydroxide, the use of which tends to accelerate corrosion and inhibit the formation of film. A. R. POWELL.

Third report on the relative corrodibilities of various commercial forms of iron and steel.

J. N. Friend (Iron Steel Inst. Carnegie Schol. Mem., 1927, **16**, 131—151 ; cf. B., 1923, 1179 A).—The results of corrosion tests on bars of different steels and irons which had been submerged in the Bristol Channel for four years showed that there is no class of ferrous material which is markedly better than others in all respects when subjected to a long-period corrosion test. Taking the average corrosion loss of wrought irons as 100, average figures for other specimens were : polished stainless steels 54, nickel-chrome steels 69, cast irons 112, carbon steels 126, Armco iron (one specimen) 152, steel (0·15% Cu, one specimen) 99. No substantial difference in pitting occurred between wrought iron and carbon steel, and stainless steel withstood the action of sea water well except where in contact with the wooden frames. Removal of the skin of cast iron by machining did not influence the rate of corrosion, but galvanising greatly prolonged the life of a single specimen of Armco iron which lost less than 50% of the amount lost by an unprotected bar. C. A. King.

Gaseous cementation of iron and steel. III. Influence of hydrocarbons on the carburisation of iron and steel. A. Bramley and G. Lawton (Iron Steel Inst. Carnegie Schol. Mem., 1927, **16**, 35—100).—In the carburisation of Armco iron in a current of carbon monoxide carrying hydrocarbon gases, *e.g.*, toluene, xylene, paraffinoid vapours, cementation was more uneven than when carbon monoxide alone was used, possibly due to irregular decomposition of the hydrocarbon in the heated zone of the furnace. Cementation by the same gases carried by an inert gas, *e.g.*, nitrogen, was even more irregular. Depth of penetration was not much affected by the rate of the gases through the furnace, but increased according to a linear law as the temperature increased ; as the period was increased in geometrical proportion the penetration increased similarly, but with a different common ratio. No indication of any migration of carbon in the carburised zone during cooling, as required by Giolitti's liquation theory, could be observed. Without exception the specimens increased in diameter and decreased in length as a result of cementation, the change in length averaging 0·0319 cm. Every deposit of carbon on the metal showed a distinct reaction for iron indicating that iron must be volatile at the temperatures (900—1100°) at which cementation was conducted. C. A. King.

Case carburisation of steels by means of salt baths of low cyanide concentration. H. B. Northrup (Trans. Amer. Soc. Steel Treat., 1927, **12**, 470—478).—Three steels of the low-carbon, nickel, and nickel-chromium types, respectively, were treated in a cyanide bath containing 23% NaCN for from 1 to 6 hrs. at 900°. Analyses of successive depths of 0·005 in. showed that the order of maximum carbon concentration, which was attained in 3 hrs. and did not exceed 0·625% C., was carbon steel, nickel-chromium steel, and nickel steel. The density of the carburised sections and maximum hardness readings were in an inverse ratio to the rapidity of carbon absorption. T. H. Burnham.

Structure and strength of cast iron. G. Neumann (Stahl u. Eisen, **47**, 1606—1609).—The tensile strength, hardness, and microstructure of grey cast iron as cast, after annealing at 850° and quenching in oil, and after quenching and annealing at 600—650° have been examined. The results confirm Bardenheuer's observations (B., 1927, 526) that the distribution of the graphite plays a far greater part in determining the mechanical properties than does any other micro-constituent. The nature of the ground mass has an effect only on the hardness, its effect on the tensile strength being masked by the action of the graphite. The highest tensile strength is obtained with the minimum amount of graphite, provided it is evenly distributed throughout the metal in the form of fine flakes. A. R. Powell.

Influence of grain size on the mechanical properties of steel, especially on the notched-bar test. H. Hanemann and R. Hinzmann (Stahl u. Eisen, 1927, **47**, 1651—1661).—The tensile strength and hardness of a plain carbon steel (0·12% C, 0·26% Si, 0·64% Mn) and of a hardened nickel–chromium steel (0·23% C, 0·26% Si, 0·38% Mn, 0·67% Cr, and 2·90% Ni) varied only slightly for grain sizes beween 600 and 13,000 μ^2 and between 90 and 25,000 μ^2, respectively. The impact test values for notched bars, however, are considerably altered by varying the grain size, although no definite relation between the two can be ascertained. For small test pieces the curve falls rapidly with increase of grain size to a constant value for medium-sized grains, then decreases rapidly above 4500 μ^2. For large test pieces the impact strength is a minimum for large and small grains and a maximum for medium sized grains. If the specimen is polished before testing, a fairly well-defined "area of flow" is discernible on the polished surface after impact ; the work done in breaking the specimen divided by the area of flow is called the "work constant" of the metal. The "work velocity" is the quotient $50A_2/A_1$, where A_1 and A_2 are the work consumed in breaking a small and a large test-piece respectively. Both the "work constant" and "work velocity" are shown to be specific properties of a metal the values of which are dependent on the grain size. Beyond a certain grain size, which varies for every metal, the value of these functions falls suddenly indicating a serious deterioration in the valuable properties of the metal. A. R. Powell.

Effect of temperature on the mechanical and microscopic properties of steel. G. C. Priester and O. E. Harder (Trans. Amer. Soc. Steel Treat., 1927, **12**, 436—445).—Tensile tests were carried out on a quenched steel (0·29% C) at high temperatures, and also after tempering at ordinary temperatures. The ratio of proportional limit to tensile strength was constant at about 50% for the high-temperature tests, but somewhat higher for the tempered specimens tested at room temperature. The reduction of area showed a rapid increase from 300° to 500° for the former conditions as compared with 200° to 400° for the quenched and tempered specimens. The microstructure showed no change up to 200°, but broke down considerably at 300°, whilst the tensile strength showed a minimum between 100° and 200° and a maximum at 300°, and the proportional limit a minimum at 100° and a maximum between 200° and 300°. T. H. Burnham.

Transverse and other tests on cast-iron test-bars. C. H. Adamson and G. S. Bell (Iron Steel Inst.

Carnegie Schol. Mem., 1927, **16**, 1—34).—Comparing actual transverse breaking stresses of cast-iron bars with calculated figures, the latter were not sufficiently accurate for practical requirements nor could the formulæ used be modified to make them more reliable. Calculated results on round bars were less than the actual results obtained, though nearer approximation was obtained with the larger diameters used. C. A. KING.

Strain in steel. Nature and detection of certain phenomena observed in permanently deformed steel. E. W. FELL (Iron Steel Inst. Carnegie Schol. Mem., 1927, **16**, 101—129).—Etchings of the surfaces of steel strained by impact and static Brinell impressions, impact of various forms of bullets, etc. have been obtained. The marked surface strains around Brinell indentations in Armco iron and mild steel were not observed with high-carbon steel and non-ferrous metals. The clearest strain-etchings figure were obtained when the temperature of straining was 200—400°; no results were observed on mild steel quenched above 700°.
C. A KING.

Effect of cold-work on the structure and hardness of single iron crystals and the changes produced by subsequent annealing. L. B. PFEIL (Iron Steel Inst. Carnegie Schol. Mem., 1927, **16**, 153—210).—From the results of deforming cubes of iron consisting in most cases of single crystals, by means of slow compression, the structure developed depends on the direction of the cold-work in relation to the orientation of the crystal, and the hardness is the same in all directions. Deformation alone does not appear to produce twinning. Lowering of the recrystallising temperature is dependent on the severity of the work applied, recrystallisation generally beginning at the grain boundaries of a banded structure, though there was no evidence of granulation or disintegration before recrystallisation. In a crystal aggregate the boundaries have a negligible effect in causing hardening; single crystals have the same hardness as the aggregate, and the Brinell impressions, which tend to become circular on a cold-worked specimen, may return to irregular shapes on the recrystallised crystal. The effect of low-temperature annealing on cold-worked iron is to cause a hardening which may be expressed as an increase in the value of n, but no change in a in Meyer's law : $P = ad^n$. C. A. KING.

Effect of varying ash in the coke on blast-furnace working. C. S. GILL (Gas World, 1927, **87**, Coking Sect., 127—128).—A series of tests on a day's run of the blast furnace using coke with varying ash content showed that the higher the ash in the coke the higher is the sulphur in the pig iron; thus with 10% of ash the iron contained 0·03% S and with 14·5% of ash, 0·2% S. More prolonged tests indicated that by using a coke with 9% of ash instead of one with 12%, the coke consumption per ton of pig iron was reduced by 1·25 cwt., the output of pig iron increased by 25%, and a more regular grade of iron was produced. A. R. POWELL.

Fundamental laws of magnetic viscosity. Influence of ageing and annealing. C. LAPP (Rev. Mét., 1927, **24**, 496—508).—Electrolytic iron exhibits the phenomenon of magnetic viscosity common to ferromagnetic substances. The viscosity is diminished by prolonged ageing at 130—180°, but at the expense of the magnetic properties of the metal; annealing at 300—500° has the inverse effect. A. R. POWELL.

Determination of cobalt and other alloyed elements in cobalt metal, cobalt steel, and high-speed tool alloys. E. SCHIFFER (Stahl u. Eisen, 1927, **47**, 1569—1571).—The determination of cobalt in the commercial metal is most satisfactorily effected by electrolysis in the usual way after removing heavy metals and iron; the deposited cobalt is subsequently tested for nickel by the dimethylglyoxime method. Equally good results are obtained by determining the impurities and taking the cobalt by difference provided that oxygen, sulphur, and included water-soluble salts are tested for. Cobalt steel with a low content of cobalt is dissolved in hydrochloric acid with the help of an oxidising agent, the iron precipitated with zinc oxide twice or as basic acetate, and the cobalt determined by means of α-nitroso-β-naphthol in the first case or by electrolysis; for rapid control work the cyanometric method gives sufficiently accurate results. For the separation of iron from cobalt in a high-cobalt steel the ether process is most satisfactory; the aqueous layer is evaporated with nitric acid to convert the metals into nitrates and the solution boiled with nitric acid and potassium chlorate to remove manganese and oxidise chromium to chromate, cobalt being subsequently separated by addition of potassium hydroxide and finally determined by electrolysis. Stellite and similar tool alloys are preferably dissolved by fusion with peroxide in a porcelain crucible; the solution is used for the determination of tungsten, molybdenum, chromium, and vanadium, and the insoluble residue for the determination of nickel, cobalt, and manganese by electrolysis. A. R. POWELL.

Presence of iodine in iron and iron slags. G. LUNDE and T. VON FELLENBERG (Z. anorg. Chem., 1927, **165**, 225—248).—Colorimetric methods are described for the accurate determination of minute quantities of iodine in silicate slags, iron, and steel. All common varieties of iron and steel contain iodine in quantities varying from about 0·1—1·10 mg./kg., the average being about 0·1—0·15 mg./kg. The average iodine content of a large variety of natural minerals, including a number of volcanic origin, is between 0·2—0·3 mg./kg.
H. F. GILLBE.

Critical temperatures in the annealing of brass wire. F. OSTERMANN (Z. Metallk., 1927, **19**, 349—351). —After annealing brass wire containing 62—64% Cu in the immediate neighbourhood of the boundary between the α and $\alpha + \beta$ regions both the tensile strength and ductility are considerably reduced and the metal tends to become brittle. This phenomenon is especially marked after a double annealing at 650°, and is due to incomplete absorption of the β crystals into α, the unabsorbed portion of the brittle β segregating along the grain boundaries of the α crystals. The most satisfactory structure of brass containing 63% Cu is obtained by annealing at 600° and cooling slowly or by annealing at 700° and quenching; in any case a second annealing operation should be avoided. A. R. POWELL.

Detection of internal stress in [brass] rods and

tubes. G. Sachs (Z. Metallk., 1927, 19, 352—357).—The method depends on the changes of shape undergone by the specimen when a small layer is trimmed off the outside. A mathematical expression is deduced from theoretical considerations connecting these changes with the internal stress in the metal. A. R. Powell.

Aluminium alloy "Aldrey." Fuchs (Z. Metallk., 1927, 19, 361—362).—The alloy "Aldrey," which has been developed for use in the manufacture of aluminium transmission lines, consists of aluminium containing 0·4% Mg, 0·5—0·6% Si, and 0·3% Fe. It has *d* 2·70, a tensile strength of 35 kg./mm.^{2}, an elongation of 6·5%, an elastic limit of 25 kg./mm.^{2}, a modulus of elasticity of 6500 kg./mm.^{2}, an electrical conductivity of 31·5 m./ohm-mm.^{2}, and a temperature coefficient of 0·0036. The necessary heat and mechanical treatment to obtain these properties comprises rolling or forging the casting, annealing within the temperature range of homogeneous solid solution, quenching in water, rolling or drawing into sheet or wire, and tempering at 140—160°. The alloy has as good a resistance to corrosion as commercial aluminium as measured by the Mylius' oxidising salt bath test. Transmission lines of this alloy after exposure to the atmosphere for two years had almost as good mechanical properties as when first prepared, showing practically complete absence of corrosion.
A. R. Powell.

Segregation phenomena in aluminium-copper alloys. W. Claus and B. Dango (Z. Metallk., 1927, 19, 358—360).—Cylinders of No. 12 alloy (92% Al, 8% Cu) cast from 680° in sand moulds were examined chemically and microscopically for evidence of segregation. In all cases the middle and outside portions contained slightly more copper than the portions midway between these zones, and usually the middle zone was somewhat richer in copper than the outside skin. This small amount of segregation was insufficient deleteriously to affect the mechanical properties of the alloy, but is interesting as being the first case in which both normal and inverse segregation occur in the same casting.
A. R. Powell.

Action of cement and gypsum on light metals. J. Meyer and K. Pukall (Chem.-Ztg., 1927, 51, 757—758).—Aluminium slowly reacts with a paste of cement or gypsum and water, but a protective coating of colloidal silicate or hydroxide forms after a time and further reaction is exceedingly slow. Magnesium under the same conditions reacts very slowly, but considerable corrosion ensues during the course of several weeks. Magnesium-aluminium alloys with a high aluminium content corrode much more slowly than either metal, but still are unsuitable for use in contact with either cement or gypsum. Calcium and its alloys with magnesium or aluminium are fairly rapidly attacked by either substance with the evolution of hydrogen.
A. R. Powell.

Type metal and lead-tin alloys. A. Travers and Houot (Rev. Mét., 1927, 24, 541—554).—The hardness of type-metal alloys containing lead, tin, and antimony increases with the tin content, the casting temperature, and the rate of cooling, the antimony content appearing to have little effect. Cast alloys may be hardened by heating at 180° for some time and cooling rapidly, but such alloys become completely annealed after keeping for seven months at the ordinary temperature. Lead containing 0—1% Sn when cast in iron moulds appears to have undergone some hardening process; after annealing at 200° the metal cannot be hardened again by heat-treatment in the solid state. The hardness of lead-tin alloys increases to a maximum with 16% Sn, and all alloys containing a preponderating amount of tin can be hardened by heat treatment in the solid state. Dilatometric curves of commercial lead show two small deviations possibly associated with allotropic changes. When pure tin is heated for prolonged periods at 500—800° the calorimetric curves subsequently obtained show a point of inflexion at 171—172° which may be due to the change from rhombohedral to tetragonal tin; no such point occurs if the metal has not been overheated.
A. R. Powell.

Chromium-plating steel articles. H. von Wartenberg (Chem.-Ztg., 1927, 51, 729).—The brittleness induced by occluded hydrogen in chromium-plated steel may be removed by heating in air at 100—200° for 60—15 min. according to the temperature. A. R. Powell.

Blast-furnace slag as ballast. Burchartz and Saenger.—See IX.

Induction furnace for small charges. Steinhaus and Kussmann. **Chromium plating.** Watts.—See XI.

Patents.

Recovery of iron from ferruginous sands, iron-ore dust, and pulverised ores. E. Fleischer (F.P. 617,079, 4.6.26. Ger., 8.6.25).—The ferruginous material is mixed with one third of its weight of carbon and with the necessary lime and alumina for fluxing purposes, the mixture is briquetted, and the briquettes are heated in a stream of producer gas and air in insufficient quantity to reoxidise the reduced iron. The spent gases are passed through a mass of bituminous coal to produce coke and a gas suitable for use in basic open-hearth furnaces. A. R. Powell.

Reduction of iron ores. Coast Range Steel, Ltd., Assees. of J. L. Avis (Can. P. 263,193, 10.2.23).—A mixture of the powdered ore, containing less than 1·5% of titania, coal, limestone, and kieselguhr, with alkali hydroxide as binding material, is briquetted and the briquettes are smelted in the usual way. A. R. Powell.

Carburising [case-hardening] iron. C. Roche and A. della Porta (F.P. 615,325, 22.9.25).—Iron articles are case-hardened in a molten bath comprising a mixture of 70% of sodium cyanide with 30% of potassium cyanide to which are added 1% of sodium and ammonium chlorides and 0·5% of sodium and potassium. A. R. Powell.

Welding rods for arc or gas welding [of steel]. F. Schulz (F.P. 617,237, 9.6.26. Ger., 10.6.25 and 15.2.26.)—The rods consist of a special steel containing at least one of the following elements: nickel, cobalt, manganese, molybdenum, vanadium, titanium, and zirconium. The welds obtained are free from occluded gas and from brittleness. A. R. Powell.

Colouring steel brown. E. Preynat (F.P. 616,849, 18.5.26).—The finished steel articles are freed from grease

and dipped into a strong solution of sodium hydroxide and carbonate containing lead carbonate, oxide, or dioxide, and heated at 135—160°. A. R. Powell.

Rail steel. J. K. Smith, Assr. to C. H. Wills (U.S.P. 1,643,321, 27.9.27. Appl., 22.6.26).—The alloy used in the manufacture of non-heat-treated steel rails contains 0·35—0·75% C, 0·5—1·1% Mn, 0·25—1·25% Cu, 0·07—1% Mo, together with the usual amounts of silicon, sulphur, and phosphorus. F. G. Crosse.

Steel alloy. L. M. Brown (U.S.P. 1,643,054, 20.9.27. Appl., 14.2.27).—The alloy contains 0·4—1·0% C, 0·5—1·5% Mn, 1·5—2·5% Si, and 0·5—1% (Mo + V) of which at least 0·15% is molybdenum or vanadium. F. G. Crosse.

Purification of cement copper. A. Wyporek (G.P. 441,740, 28.3.25).—Crude cement copper is digested with an excess of hydrochloric acid over that required to dissolve the basic salts present, and the solution is diluted with twice its volume of water and treated with clean scrap iron to reduce any dissolved cuprous chloride. The precipitate is collected, washed with hot water to remove chlorides, dried, and smelted. A. R. Powell.

Cementation of copper, nickel, or their alloys. Établ. de Dion-Bouton, Soc. Anon. (F.P. 616,121, 12.5.26).—The metal is heated for 6 hrs. at 750° in a mixture of finely-divided aluminium (80 pts.) and copper or nickel (20 pts.) together with some ammonium chloride. The resulting layer of aluminium bronze has a high tensile strength and is very resistant to oxidising action. A. R. Powell.

Deoxidation of copper and its alloys. Siemens & Halske A.-G., Assees. of G. Masing (G.P. 441,930, 25.3.23).—Molten copper is treated with a deoxidising agent, such as phosphorus, which is soluble in the liquid metal, then with a second substance, *e.g.*, calcium, which is also soluble in the molten metal, but which forms a compound with the excess of the deoxidising agent, which is difficultly soluble in molten copper and which forms no solid solution or alloy with solid copper. A. R. Powell.

Precipitating copper from sulphate solution. N. C. Christensen (U.S.P. 1,643,922, 27.9.27. Appl., 10.3.24).—The sulphate solution is treated with metallic lead, the coating of lead sulphate formed upon the lead being continuously removed by mechanical means; the mixture of copper and lead sulphate is afterwards separated by the action of a differential solvent. M. E. Nottage.

Treatment of scrap containing gunmetal and white metal in order to separate the latter. J. Spence, C. E. Courtenay, and H. A. Courtenay (E.P. 276,730, 31.5.26).—The scrap metal is placed on a wire-gauze tray supported in a dome-shaped chamber, heated by means of hot furnace gases. The metal is heated at a temperature above the m.p. of the white metal, and the tray is then vibrated so that the molten metal drains off through the bottom into a suitable receptacle. M. E. Nottage.

Recovery of nickel from silicate ores. T. Tatebe (F.P. 617,033, 3.6.26).—Finely-powdered garnierite is reduced with carbon or in a current of hydrogen or carbon monoxide at 500—800°, and the product is leached with ammoniacal ammonium carbonate solution, preferably in the presence of an oxidising agent such as manganese dioxide or a chlorate. The filtered solution is either directly electrolysed for nickel or treated in such a way as to precipitate nickel hydroxide or carbonate which is reduced to metal in the usual way. A. R. Powell.

[Nickel-molybdenum-chromium] alloy. T. D. Kelly (E.P. 277,170, 10.9.26).—A malleable alloy melted under the protection of a flux consisting of a mixture of calcium or magnesium chlorides and carbon contains 33·3—90 pts. of nickel, 5—33·3 pts. of molybdenum, and 5—33·3 pts. of chromium. C. A. King.

Non-magnetic alloy of high electrical resistance. Y. Kamishima (E.P. 276,874, 3.1.27).—The alloy contains 9—12% Ni, 15—20% Cr, 0·3—2% Mn, and remainder chiefly iron; it must contain no silicon, and the carbon content should be as low as possible. M. E. Nottage.

Manufacture of chemically pure aluminium. Deuts. Versuchsanstalt für Luftfahrt, E.V. (E.P. 276,911, 6.4.27. Ger., 2.12.26).—In the electrolytic purification of aluminium, precious metals (*e.g.*, gold, platinum, etc.) of high sp. gr. which are not taken up in solid solution by aluminium are added to aluminium of 99·8—99·9% purity for the purpose of increasing the density of the anode layer, thereby making this the lowermost layer, whilst the purified aluminium forms the uppermost and cathode layer. M. E. Nottage.

Metallurgy of readily oxidisable metals, *e.g.*, magnesium. G. Michel (E.P. 271,088, 11.5.27. Fr., 11.5.26).—During melting, casting, or other use of easily oxidisable metals, *e.g.*, magnesium, the metal is protected from oxidation by supplying an atmosphere of sulphur dioxide over the surface of the molten metal. C. A. King.

Lead alloys. H. Yoshikawa (E.P. 277,199, 22.11.26).—A lead alloy contains less than 10% of a metal immiscible with lead and less than 4% Bi, or preferably less than 2% Cu (1% Ni) and less than 1% Bi. C. A. King.

[Lead-antimony-copper] alloy. C. A. Geatty (U.S.P. 1,644,425, 4.10.27. Appl., 14.4.25).—An alloy for lining brake bands etc. consists of 95% Pb, 3% Sb, and 2% Cu. F. G. Crosse.

Process of purifying cadmium. H. Howard, Assr. to Grasselli Chemical Co. (U.S.P. 1,644,431, 4.10.27. Appl., 24.4.22).—Cadmium sponge containing zinc as impurity is purified by boiling with a solution of caustic soda (d 1·45). F. G. Crosse.

Concentration of tin minerals. W. O. Snelling (U.S.P. 1,638,279, 9.8.27. Appl., 31.10.25).—Cassiterite concentrate is treated electrolytically or with zinc and hydrochloric acid to form on the particles a surface layer of tin, and thus to permit of their ready separation by flotation or by passage over amalgamated plates. T. S. Wheeler.

Treatment of tin ores. H. L. Sulman and H. F. K. Picard (E.P. 276,743, 2.6. and 13.12.26 and 24.2.27).—Low-grade tin concentrates containing iron, copper, tungsten, etc. are mixed with bituminous coal and the mixture is formed into briquettes which are coked at

600—700° in a cast iron retort lined with acid-resisting enamel. Hydrogen chloride is then passed through the mass to convert the tin into stannous chloride which is collected in a vessel heated at 130°, the excess hydrogen chloride being subsequently collected separately. The stannous chloride remaining in the retort may be substantially recovered by blowing in a limited quantity of steam which converts the chlorides of iron and copper into oxides. In the first treatment 80% of the tin is recovered and a further 15% in the second treatment. The stannous chloride may be converted into metal by electrolysis or other suitable process. A. R. POWELL.

Coating metals with metal. E. C. R. MARKS. From AMER. MACHINE & FOUNDRY CO. (E.P. 276,390, 23.3.26).—Metal to be coated is passed continuously through a chamber in which the vapour of the coating metal is produced and maintained under relatively low pressure, *e.g.*, 10 in. of mercury, and high electric potential, *e.g.*, 2000 volts. The atmosphere should be perfectly dry. An air blast is directed towards the coated metal as it leaves the chamber, and a similar blast, preferably carrying sand for cleaning purposes, may impinge on the metal before entering. C. A. KING.

Production of aluminium coatings on metals. METALLISATOR BERLIN, A.-G. (G.P. 441,964, 16.11.24).—The articles are coated with a mixture of aluminium or magnesium powder, powdered silicon, or a silicon compound, and an inorganic binder. After drying, the articles are heated to cause the aluminium to alloy with the base metal, in which operation the presence of silicon acts as an accelerator. A. R. POWELL.

Provision of protective layers on reflecting bodies. "METALLIQUE" and "DURAL" (E.P. 249,122, 10.3.26. Czecho-Slov., 11.3.25).—A protective layer of water-glass, with the addition of about 25% of caustic potash liquor is applied to mirrors, specula, etc. between the reflecting layer and the metallic coating which is applied by the metal-spraying process. W. G. CAREY.

Process of reacting [oxidising] metals with gases. H. R. CARVETH, Assr. to ROESSLER & HASSLACHER CHEMICAL CO. (U.S.P. 1,638,471, 9.8.27. Appl., 9.3.23).—The metal, *e.g.*, zinc, is mixed with its oxide (20 pts.), and the powdered mass is treated with air at a temperature between the m.p. of the metal and that of the oxide. T. S. WHEELER.

Reduction process using circulating gases. E. EDWIN, Assr. to A./S. NORSK STAAL ELEKTRISK-GAS-REDUKTION (U.S.P. 1,638,684, 9.8.27. Appl., 15.10.25. Norw., 27.9.22; cf. F.P. 596,714; B., 1926, 231).—The efficiency of processes involving the reduction of iron ore by circulating producer gas is increased by treating a portion of the gas with steam at 400° to form carbon dioxide and hydrogen, removing carbon dioxide with water under pressure, and returning hydrogen to the circulation system. T. S. WHEELER.

Process for decomposing insoluble minerals. G. BERGE, Assr. to H. SPURRIER (U.S.P. 1,642,667, 20.9.27. Appl., 4.12.25).—The mineral is treated with boric acid at a temperature sufficient to convert certain of its basic constituents into borates. The latter are then treated with dilute nitric acid to produce the nitrates and free boric acid. H. HOLMES.

Apparatus for determining hardness [of metals] by means of a spherical impression. L. SCHOPPER (G.P. 441,832, 2.5.25).—In an apparatus for determining the hardness of metals on the Brinell principle the carrier for the pressure arm and the pressure gauge comprises a closed framework of one piece of metal inside which the gauge is screwed. A. R. POWELL.

Moulding sand. W. STOLL (G.P. 440,402, 26.9.25).—Moulding sand for the production of non-porous castings comprises a mixture of 92% of powdered pumice, 3% of silica, and 5% of powdered sugar. A. R. POWELL.

Chromium plating machine. W. S. EATON (E.P. 276,921, 2.5.27).—A chromium plating machine comprises adjacent insulated tanks containing, respectively, a cleaning and a plating bath, permitting convenient transfer from one tank to another, a housing having a closed top, and an opening at the front provided with closing means, anodes carried by a bus bar, and a suction device communicating with the housing.
J. S. G. THOMAS.

Manufacture of edge tools and special composition of steel for same. A. E. G. T. VON VEGESACK (U.S.P. 1,644,097, 4.10.27. Appl., 2.8.26. Swed., 26.9.25).—See E.P. 268,616; B., 1927, 448.

Coating iron or its alloys with metals. A. STRASSER (E.P. 255,417, 28.4.26. Ger., 17.7.25).—See F.P. 607,381; B., 1927, 194.

Purifying or refining metals and alloys. D. R. TULLIS (U.S.P. 1,636,881, 26.7.27. Appl., 9.11.26. U.K., 11.11.25).—See E.P. 272,336; B., 1927, 606.

Production of high-grade silicon-iron. V. B. BROWNE (E.P. 277,537, 10.1.27).—See U.S.P. 1,570,229; B., 1926, 282.

Hermetically closing the annealing chambers of annealing furnaces. SIEMENS-SCHUCKERTWERKE G.M.B.H. (E.P. 263,162, 15.12.26. Ger., 18.12.25).

Apparatus for producing oxyacetylene mixtures for use in spraying metals and other fusible material. W. CARPMAEL. From SOC. ITAL. PER LA METALLIZZAZIONE (E.P. 277,186, 14.10.26).

[Rotary transporter for plant used in] the detinning of tinned iron scrap etc. M. A. ADAM (E.P. 277,127, 16.6.26).

Apparatus for pickling tubes, bars, etc. WELLMAN SEAVER ROLLING MILL CO., LTD., and S. SMITH (E.P. 276,732, 1.6.26).

Separation of ores (E.P. 277,210).—See XI.

XI.—ELECTROTECHNICS.

High-frequency [induction] furnaces for small charges. W. STEINHAUS and A. KUSSMANN (Z. Metallk., 1927, **19**, 346—348).—The current supply for a small (5 kw.-hr.) induction furnace is obtained by transforming up the ordinary A.C. supply to 6000 volts by means of a suitable transformer in the primary circuit of which is arranged an ammeter, choking coil, and cut-out switch. The high-tension current thus obtained is shunted through three oil condensers in parallel, and converted to high frequency by means of a spark gap device consisting of a rotating aluminium disc, 25 cm.

in diam. and 4·5 mm. thick, on both sides of which are arranged twelve equally spaced projections which allow the passage of current to two stationary electrodes as the disc rotates. The projections consist of copper blocks tapering to small discs 4·5 mm. in diam., and the stationary electrodes are somewhat similar in shape, but are hollow inside for water-cooling. The best results are obtained with 3000—3500 revolutions of the disc per min. The heating coil of the furnace consists of 27 turns of flat, water-cooled copper tube, has an internal diameter of 6 cm., and is 15 cm. high. Temperatures up to 1800° can be readily obtained with suitable charges. A. R. Powell.

Anodes for chromium plating. O. P. Watts (Amer. Electrochem. Soc., Sept., 1927. Advance copy. 9 pp.).—Small-scale experiments have been made to test the effect of using various anode materials, including chromium, lead, iron, steel, and various chromium and silicon alloys, in a chromium plating bath. Under the conditions of experiment lead anodes were most satisfactory in that good cathode deposits were obtained at high efficiency. Iron and steel were found to possess several disadvantages resulting from accumulation of iron salts in the solution. Iron–silicon alloys may be slightly more satisfactory. H. J. T. Ellingham.

Electrolytic preparation of *p*-aminophenol. D. Caeser (Amer. Electrochem. Soc., Sept., 1927. Advance copy. 13 pp.).—Experiments have been made to determine the most suitable electrode materials for use in the electrolytic preparation of *p*-aminophenol using a cell in which the catholyte, contained in a porous cup, is a solution of 20 g. of nitrobenzene in 150 g. of concentrated sulphuric acid to which a few drops of water have been added, whilst the anolyte is 96% sulphuric acid. Platinum, carbon, cobalt, and copper gauze were tried as cathode materials, and platinum, lead, carbon, duriron, ferro-chromium, and lead coated with manganese dioxide as anode materials. Of those giving satisfactory results, a copper gauze cathode and a duriron anode are considered most suitable for technical use. Operating at 70—80° with this combination, the maximum yield of *p*-aminophenol (41—42%) is obtained when the cathodic current density is between 3·8 and 5·1 amp./dm.^{2} With 4 amp./dm.^{2} the current efficiency was 39·3%, the bath voltage being about 4 volts. Further possibilities of improvement and the technical advantages of the electrolytic method are discussed. H. J. T. Ellingham.

Electrodialysis in biochemistry. Technical processes. J. Reitstötter (Kolloid-Z., 1927, 43, 35—41; cf. Dhéré, A., 1927, 423).—A bibliography is given of the patent literature dealing with electrodialysis, and examples are drawn from the literature to demonstrate the fact that the process of electrodialysis was known and applied to the elucidation of technical problems some years before the mechanism of the process was understood. Special reference is made to the work of Schwerin, who, it is shown, recognised the nature of the process in 1900, although the term "electrodialysis" was only used much later by Pauli's school. A detailed description is given of the apparatus used by Schwerin for the extraction and purification of syrups, which satisfies Dhéré's conception of electrodialysis.
L. L. Bircumshaw.

Magnetic viscosity. Lapp. **Aluminium alloy "Aldrey."** Fuchs.—See X.

Electrodeposition of rubber. Sheppard.—See XIV.

Patents.

Apparatus for electrolysing solutions of alkali chlorides. E. Krebs (E.P. 267,560, 14.3.27. Ger., 15.3.26).—An electrolysing unit consisting of a cathode, diaphragm, anode, and cell top is arranged in an open trough or channel extending along the upper edges of all four sides of the container, and acting as a water or other liquid seal. A flange projecting from the electrolysing unit rests on the bottom of the trough and supports the unit. J. S. G. Thomas.

Cell with mercury cathode for the decomposition of alkali chlorides. F. Gerlach (E.P. 269,513, 30.3.27. Ger., 17.4.26. Cf. F.P. 596,918; B., 1926, 284).—An electrolytic cell for the decomposition of alkali chlorides comprises a mercury cathode, an amalgam-formation chamber lined with polishable stone or similar material, *e.g.*, granite, and in which the distance apart of the electrodes may be made very small without risk of a short-circuit occurring, and a separate amalgam-decomposition chamber situated at a lower level. Transfer of mercury from the second chamber to the first is effected without shock by means of a worm having a horizontal disc which distributes the mercury below the surface of a covering liquid. J. S. G. Thomas.

Electrolytic cell [for electrolysis of water]. A. T. Stuart (E.P. 254,268, 13.4.26. Can., 27.6.25).—A sheet-metal electrode of one polarity is placed between two sheet-metal electrodes of the opposite polarity so that its major surface lies in the same direction as the current across the spaces between the electrodes. [Statutory Ref. to E.P. 215,355.] J. S. G. Thomas.

Apparatus for the electromagnetic separation of ores. A. Davies (E.P. 277,210, 8.6.26).—In an electromagnetic separator of the kind described in E.P. 105,354 (B., 1917, 600), the point or points of the upper lifting magnets are formed so as to produce an interrupted attraction on the material to be treated.
J. S. G. Thomas.

Electrical precipitation and separation of suspended matter in gases and non-conducting liquids. Metallbank u. Metallurgische Ges. A.-G. (G.P. 441,912, 29.4.25).—An apparatus for removing suspended matter from gases and non-conducting liquids comprises a vessel carrying longitudinal, unsymmetrical discharge electrodes unsymmetrically arrranged between pairs of precipitation electrodes. A. R. Powell.

Apparatus for treating gases and vapours with the silent electric discharge. I. G. Farbenind. A.-G., Assees. of W. J. Müller and O. Nitzschke (G.P. 442,412, 30.9.25).—The inner tube is loose within the outer tube, so that the distance between the two tubes can be adjusted below by prominences or cones on the inner or outer tubes, and above by depressions on the outer tube or prominences on the inner tube or other distance pieces. A number of tubes are arranged in a watertight manner in a chamber serving as cooler, and gas or vapour is led into the tubes above the water level and, after treatment, passes to a collecting funnel.
J. S. G. Thomas.

Electric furnace. F. A. J. FITZGERALD, Assr. to HARPER ELECTRIC FURNACE CORP. (U.S.P. 1,643,808, 27.9.27. Appl., 1.6.25).—An electric furnace comprises a resistor extending through and radiating heat downwards in a chamber arranged in the upper part of a casing, means for conveying articles to be treated in the lower part of the casing, and a heat-distributing structure for heating articles on this conveyor.
J. S. G. THOMAS.

Electric furnace. A. D. KEENE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,643,774, 27.9.27. Appl., 10.2.25).—An electric resistor is supported by, and in spaced relation with, a muffle enclosed within a furnace chamber surrounded by refractory walls. J. S. G. THOMAS.

Electric furnace. I. RENNERFELT (E.P. 276,823, 24.8.26).—The flat contacting faces of the refractory sections of the furnace are arranged at right angles to the longitudinal axis of the furnace chamber, and each section carries one or more metallic heating elements in the form of ribbon, wire, or coil which, in part, are arranged round the longitudinal axis close along the inner wall of the section, the ends being brought out at right angles to the axis. J. S. G. THOMAS.

Electric accumulator. K. NEMETH and A. SZANTO (E.P. 276,797, 20.7.26).—The cathode is made of a sieve-like alloy of zinc, nickel, and copper, *e.g.*, of Argentan, amalgamated and covered with zinc. Sulphuric acid free from zinc is used as electrolyte.
J. S. G. THOMAS.

Dry cell. A. HEIL (F.P. 615,578, 4.5.26. Ger., 15.2.26). —The carbon electrode is surrounded to two thirds or three quarters of its height by a depolariser consisting of a mixture of natural or artificial manganese dioxide, graphite, and lampblack, or a mixture of the last two with finely-powdered ammonium chloride. Carton paper is arranged between the zinc cylinder and the other components of the cell. J. S. G. THOMAS.

Electrolytic coppering of the outer carbon face of lamellar dry batteries having a carbon electrode. ČESKÁ ZBROJOVKA AKC. SPOL. V. PRAZE (Swiss P. 118,083, 17.10.25. Czecho-Slov., 15.11.24).—A cell comprising electrolyte between plane zinc and carbon electrodes is filled, sealed, and placed vertically in the coppering bath so that a current flows between the carbon electrode and an auxiliary electrode immersed in the bath.
J. S. G. THOMAS.

Manufacture of incandescence filaments. COMP. DES LAMPES (F.P. 614,553, 15.4.26. U.S., 17.4.25).—Tungstic oxide, mixed with small proportions of aqueous solutions of water-glass and potassium chloride or similar substances containing an alkali metal and silicon, is treated with hydrochloric acid, reduced with hydrogen, and the resulting product worked up into filaments having large overlapping and coalescing crystals. When formed into helices of very small diameter, such filaments do not sag and are very resistant to shocks.
J. S. G. THOMAS.

Material introduced into electric incandescence lamps and similar glass vessels [to prevent blackening]. PATENT-TREUHAND GES. FÜR ELEKTRISCHE GLÜHLAMPEN M.B.H. (G.P. 441,865, 8.11.25).—A compound of phosphorus oxide with the oxide of any metalloid. *E.g.*, boron phosphate is introduced into the vessel and combines with any water vapour which may be present or formed. J. S. G. THOMAS.

Electron discharge device. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of I. LANGMUIR (E.P. 267,504, 5.3.27. U.S., 10.3.26).—In an electron discharge device, means are provided for trapping positive ions in the space surrounding the cathode and for producing an electric or magnetic field for varying the electron current thereby freed from space-charge limitation.
J. S. G. THOMAS.

Thermionic valve. EDISON SWAN ELECTRIC CO., LTD., L. H. SOUNDY, and T. W. PRICE (E.P. 276,703, 28.4.26).—Unequal emission of electrons from different parts of a filamentary cathode heated by an electric current is reduced by coating a very small fraction, *e.g.*, less than one tenth of the length of the cathode, with a substance, *e.g.*, a paste of calcium and strontium oxides, having high electron emission properties.
J. S. G. THOMAS.

Electrical deposition of organic materials [*e.g.*, rubber, cellulose, etc.]. KODAK, LTD., Assees. of S. E. SHEPPARD and C. L. BEAL (E.P. 251,271, 20.4.26. U.S., 22.4.25).—See U.S.P. 1,589,326; B., 1926, 793.

Gas-filled incandescence lamp and getter therefor. D. S. GUSTIN, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,643,810, 27.9.27. Appl., 16.5.23).—See Swiss P. 114,997; B., 1927, 257.

Apparatus for analysis (E.P. 252,206).—See I.

Non-magnetic alloy (E.P. 276,874). **Coating metals** (E.P. 276,390).—See X.

Rubber goods (E.P. 269,505).—See XIV.

XII.—FATS; OILS; WAXES.

Addition of bromine to butter and margarine fat. A. SCHNECK (Milch. Zentr., 1927, 56, 181—183).—Hehner's method for the determination of the bromine absorption of butter and margarine fats gives results which, when multiplied by the factor 1·5879, are in good agreement with the iodine values obtained by the Hübl method. The drying of the bromo-compound to constant weight should be continued until two consecutive weighings differ by no more than 0·5 mg., which for fat portions of about 1·3 g. is equivalent to 1 unit in the first decimal place of the iodine value. The increase of refractive index on bromination of these fats is proportional to the weight of bromine added, and hence the iodine value may be calculated from the difference in the refractive indices of the fat and of its completely brominated derivative. Using a Zeiss butyrometer reading to 0·1 scale divisions, the limit of accuracy for the iodine value is 2 units in the first decimal place.
F. R. ENNOS.

Determination of the acetyl value [of fats]. F. CRONER (Z. angew. Chem., 1927, 40, 1013—1014).—About 2 g. of the acetylated product is dissolved in 5 c.c. of neutralised alcohol and titrated in the cold with 0·5N-alcoholic potassium hydroxide; a further 25 c.c. of the same solution are added and the mixture is heated for 30 min. on the water-bath until the substance

is completely saponified. After evaporating off the alcohol the residue is dissolved in water and the solution transferred to a long-necked Kjeldahl flask fitted with a bulb trap. An excess of sulphuric acid is added and the liberated acetic acid distilled with steam into an Erlenmeyer flask previously rinsed with neutralised alcohol. When 600—700 c.c. have been collected the distillate is titrated with $0 \cdot 1N$-potassium hydroxide using phenolphthalein as indicator. The use of the Kjeldahl flask and trap avoids errors due to sulphuric acid spray being carried over with the distillate.

A. R. Powell.

Colorimetric determination of minute quantities of dissolved phosphorus in oil. C. Stich (Z. angew. Chem., 1927, 40, 1014).—A standard phosphorus solution is prepared by dissolving 0·1 g. of yellow phosphorus in 100 g. of a fatty oil containing 2% of ether or 1% of terpene and further diluting 10 g. of this solution with 90 g. of the oil so that the standard solution contains 0·1 mg. of phosphorus per g. A series of standard tubes is then prepared containing 0·1—1 g. of the oil, diluted to 10 c.c. with a mixture of ether, absolute alcohol, and acetone in the volume ratio of 40 : 20 : 5 ; to every tube are added 10 drops of a saturated solution of silver nitrate in acetone, which produces a brown colour the intensity of which is proportional to the phosphorus content. The standard solutions so prepared are stable for long periods if protected from moisture and air, provided that the reagents used are absolutely anhydrous. To carry out a determination on an unknown oil, 1 g. is diluted to 10 c.c. with the ether-alcohol mixture, silver nitrate in acetone is added, and the colour compared after 10—15 min. with the series of standards.

A. R. Powell.

Water in oils. Pflug.—See II.

Patents.

Stable saponin solution of good cleansing action. A. Christen (Swiss P. 117,310, 26.8.25)—The stability and cleansing power of a saponin solution are improved by stirring in 1·5—2% of free acid and a little potassium nitrate in the cold.

S. S. Woolf.

Deodorisation, clarification, and neutralisation of vegetable oils. E. Foray (Addn. No. 31,289, 17.10.25, to F.P. 601,919 ; B., 1926, 987).—After neutralisation with an amount of alkaline solution determined by preliminary titration, the oil is filtered, washed, and subsequently decolorised and deodorised by carbon, according to the earlier patent.

S. S. Woolf.

Manufacture of a finely-divided dry soap product. R. L. Holliday, Assr. to Industrial Spray-Drying Corp. (Re-issue 16,749, 27.9.27, of U.S.P. 1,621,506, 22.3.27).—See B., 1927, 339.

Light hydrocarbons from oils (E.P. 263,082 and 276,007). **Bituminous emulsions** (E.P. 276,543). —See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Paint tests on wood. H. A. Gardner (Amer. Paint and Varnish Manufrs.' Assoc., Aug., 1927, Circ. No. 315, 439—446).—A preliminary account of exposure tests now proceeding in Florida and Washington, physical properties of the paints under test being tabulated. The effect of introducing heavy-bodied oil or varnish into paints for exterior use in order to prevent chalking etc. is being studied.

S. S. Woolf.

Are there resinogenic and resinophoric groups? H. Wolff (Z. angew. Chem., 1927, 40, 1010—1011).—A discussion of the nature of resins and of the possibility of the existence of resinophoric groups and resinogenic groups. As the chemical composition and physical characteristics of resins vary to a considerable extent it appears to be impossible to postulate the existence of any one group common to all resins or to any class of resins. On the other hand there are many groups which are capable of reacting in such a manner as to produce colloidal substances of high mol. wt., and these may perhaps be considered as resinogenic groups.

A. R Powell.

Durability of resins and plasticisers in lacquers. H. A. Gardner and A. W. van Heuckeroth (Amer. Paint & Varnish Manufrs.' Assoc., Aug., 1927, Circ. No. 316, 447—468).—Exposure tests on 18 resin solutions alone, extended with oil, or incorporated with a standard lacquer base, clear and pigmented, are detailed. The conditions of the films at time of failure are described or, in suitable cases, reproduced by photomicrographs. Exposure tests on a parallel series of lacquers containing 21 different plasticisers are also described, the mechanical properties of stripped films of these lacquers after 24 hrs., 48 hrs., and 1 week being tabulated. Further tests were carried out on a lacquer containing shellac (for which a special lacquer formulation was necessary).

S. S. Woolf.

Patents.

Liquid coating composition. E. C. R. Marks. From Bakelite Corp. (E.P. 276,417, 20.5.26).—A phenol–formaldehyde resin in the initial stage of condensation is dissolved in an aqueous alkaline solvent, and, as hardening agent and diluent, a liquid aldehyde having a higher mol wt. and lower reactivity than formaldehyde (*e.g.*, furfuraldehyde) is added.

S. S. Woolf.

Moulding phenolic condensation products. H. C. Heide. From Products Protection Corp. (E.P. 276,440—1, [a, b] 28.5.26).—(a) After desiccation, phenol-aldehyde condensation products in the initial stage are heated throughout at a temperature slightly below that of transition to the intermediate stage. They are then introduced into heated moulds, subjected to suitable pressure and temperature to effect the final condensation, and allowed to cool gradually, the homogeneity etc. of the product being improved. (b) The "curing" process is carried out by submerging the moulding in a liquid dielectric of greater viscosity than water, *e.g.*, "cumar," chlorinated naphthalenes, etc., and subjecting liquid and moulding to heat and pressure in suitable apparatus.

S. S. Woolf.

Manufacture of artificial resins. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 276,518, 4.10.26).—Artificial resins obtained by the condensation of mixtures of cyclic ketones or of mixtures of these with aromatic or hydroaromatic hydroxy-compounds in the presence of condensing agents, may be used as such

or in conjunction with other artificial or natural resins, cellulose esters, rubber, etc. S. S. WOOLF.

Solutions of cellulose esters [for use as lacquers]. J. G. DAVIDSON, Assr. to CARBON & CARBIDE CHEMICALS CORP. (U.S.P. 1,644,417—1,644,420, 4.10.27. Appl., [A] 13.6.24, [B] 20.7.25, [C] 5.8.25, [D] 16.8.27).—See E.P. 238,485; B., 1925, 857.

Method of improving aldehyde resins. W. O. HERRMANN, H. DEUTSCH, and W. HAEHNEL, Assrs. to CONSORTIUM FÜR ELEKTROCHEM. IND. M.B.H. (U.S.P. 1,643,496, 27.9.27. Appl., 26.7.23. Ger., 8.7.22).—See E.P. 201,916; B., 1925, 45.

Manufacture of condensation products from phenols and formaldehydes. E. J. P. C. DE JARNY (U.S.P. 1,643,447, 27.9.27. Appl., 6.4.26. Fr., 16.4.25).—E.P. 265,033; B., 1927, 259.

Dispersing solids in liquids (E.P. 276,400—1 and 276,727—8).—See I.

Condensation products of aldehydes with ketones (E.P. 264,830).—See XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Electrodeposition of rubber. S. E. SHEPPARD (Amer. Electrochem. Soc., Sept., 1927. Advance copy. 35 pp.; cf. B., 1925, 681).—The relevant properties of rubber latex and the general principles of cataphoresis are reviewed. The particles in rubber latex have a diameter of about 2μ, carry a negative charge corresponding to a contact potential of $-0 \cdot 035$ volt, and have a mobility of about $2 \cdot 7 \times 10^{-4}$ cm./sec. under unit potential gradient, a value comparable with those for particles in most suspensions and emulsions. The relation $n \propto c/\kappa$, where n is the amount of rubber transported towards the anode per unit of current, c the concentration of rubber in the latex, and κ the total conductivity of the latter (cf. Laing, A., 1923, i, 937), is obeyed approximately, but not exactly. At the anode simultaneous discharge of hydroxyl ions, which may lead to oxidation or production of a porous structure due to oxygen evolution, can be avoided (1) by dissolving in the latex depolarisers such as pyrogallol or substances furnishing ions such as the hydrosulphide, polysulphide, or thiosulphate ions which are more easily discharged than hydroxyl ions and yield desirable products (cf. B., 1926, 639), or (2) by using attackable anodes, such as zinc, which form oxides which are desirable constituents of the deposit. Hollow carbon anodes have the advantage that they can be made porous, so that anodic gases can be pumped off or depolarisers passed under suitable pressure through the anode (cf. B., 1926, 598). The freshly-deposited rubber is evidently semi-permeable, for continuous deposition of thick layers is possible; the mechanism of this process is discussed. Moreover, if the anode is surrounded at some distance by a porous diaphragm, rubber is deposited on the diaphragm, a process in which the action of anodic products on the rubber is avoided, and one which can be applied to the impregnation or coating of fabrics (B., 1924, 104; 1925, 46). Substances such as sulphur and various fillers, pigments, and softeners, which can be obtained in the form of negatively-charged suspensions, form a stable "mix" with the latex, and are electrodeposited with the rubber in approximately the same proportions as in the mix (cf. B., 1925, 681). These "wet mixes" are easy to prepare and handle, and give homogeneous deposits, for the small differences between the mobilities of the different kinds of particles seem to be reduced in such mixtures, particularly when the total concentration is high. Szegvari's theory of the mechanism of the deposition and the influence of other anodic processes on it is discussed. With a latex containing 7 g. of rubber per 100 c.c., 1 faraday deposits about 1500 g. of rubber. There is no necessary limit to the thickness which can be deposited, but practically it is limited by the question of drying and by secondary changes in the surface layers. The amount of water in the solidified layer may be from 25 to 50%, depending on the conditions of deposition. The electric gradient in the deposit affects the texture, and a "natural" graining effect may be produced. The throwing power is not very high, but higher than would be expected from the low conductivity of the bath, and is generally sufficient to deal with surfaces of irregular shape. Rubber produced by electrodeposition may be vulcanised readily, and permits the use of "super-accelerators" which cannot be used in milled stock, since they act so rapidly as to produce scorching. Owing to the absence of milling, the tensile strength and elasticity of electrodeposited rubber are extremely high, and in the absence of "ageing" compounds it is superior to ordinary milled rubber in ageing properties; ageing compounds are easily introduced and utilised. Hygroscopic substances are readily washed out of the deposit, so that good insulating properties can be secured.

H. J. T. ELLINGHAM.

Variation of Ceylon estate grades of rubber. REP. LONDON COMM. CEYLON RUBBER RES. SCHEME (Bull. Imp. Inst., 1927, **25**, 218—242).—Examination of 39 samples of Ceylon plantation rubber indicates that machine-dried blanket crêpe rubber is most plastic and least variable in this feature. Smoked sheet is generally fairly soft, but more variable, whereas air-dried crêpe is frequently tougher than the other two. There is a distinct relationship between plasticity and rate of vulcanisation, softer rubbers generally having a low rate of vulcanisation, whilst tougher rubbers are more rapid; this relationship is probably connected with maturation of the rubber in the interval between coagulation and completion of drying. When mixed with sulphur, the machine-dried crêpe rubber gives the highest degree of uniformity in rate of vulcanisation; in the additional presence of zinc oxide and an organic accelerator, none of the three varieties of rubber shows any important variation in vulcanising or mechanical properties. From a comparison of plasticity measurements made by the parallel plate method and the extrusion method, respectively, it is shown that the latter is more sensitive, and so more useful for exact investigations. D. F. TWISS.

PATENTS.

Caoutchouc-latex compositions. A. BIDDLE (E.P. 277,194, 10.11.26).—Casein as such or in the form of material containing casein, *e.g.*, skimmed milk, is introduced in substantial proportion into rubber latex. The mixture may be precipitated by the addition of acid.

D. F. TWISS.

Manufacture of rubber goods from rubber dispersions by electrophoretic means. ANODE RUBBER CO., LTD. (E.P. 269,505, 21.3.27. Ger., 14.4.26).—In order to prevent the liberation of gases at the cathode during the electrophoretic deposition of rubber from rubber dispersions, an oxidising depolarising agent is provided; the cathode, for example, may be of lead coated with lead dioxide. Hollow articles such as air-tubes may be produced on the inner surface of a sub-divided annular anode which acts as the container, the cathode consisting of an annular lead-covered iron tube with a surface coating of lead dioxide. D. F. TWISS.

[Accelerators for the] vulcanisation of rubber. W. SCOTT, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,638,220, 9.8.27. Appl., 23.4.24).—A primary arylamine, *e.g.*, aniline (1 mol.), is heated at about 80° with acetaldehyde or a higher homologue (1·5 mols.), and the condensation product is treated with 40% formaldehyde solution. T. S. WHEELER.

Production of rubber mixtures. METALLBANK U. METALLURGISCHE GES. A.-G., Assees. of F. MILDNER (G.P. 441,383, 9.7.25).—The latex is worked up to a paste with a solvent or swelling agent, softening agents being added if desired, and fillers and vulcanisers are added dry to the paste. L. A. COLES.

Protection of metal (E.P. 276,705).—See X.

XV.—LEATHER; GLUE.

Theory of tanning. Y. H. LI (J. Amer. Leather Chem. Assoc., 1927, **22**, 380—444).—Tanning tests with naphthalene and triphenylcarbinol derivatives, phenols, and benzene derivatives containing no hydroxyl groups show that the hydroxyl group is the primary tanning group for certain classes of compounds, *e.g.*, naphthalene and triphenylcarbinol derivatives. This group is present in the original compound in the naphthalene derivatives, but may be formed during tannage with triphenylcarbinol derivatives. The hydroxyl group must be in the α-position for the naphthalene derivatives to possess tanning properties, and in the form of a carbinol group in triphenylmethane derivatives, which agrees with Mathur's findings on the tanning properties of fatty acids (B., 1927, 284). The tanning properties of α-naphthol derivatives may be modified by the entering groups, especially acid and basic groups, but are not entirely nullified by them. Tanning with certain aromatic hydroxy-compounds is essentially a chemical reaction between the hydroxyl group of the naphthol and the hide substance, with the elimination of water. The reaction may be more complete so that all the hydrogen atoms in the amino-group are replaced by tanning materials. D. WOODROFFE.

Treatment of red gum or marri kino (*Eucalyptus calophylla*) for the preparation of tannin extract. (Austral. Council for Sci. & Ind. Res., 1927, Circ. No. 9, 14 pp. Cf. Salt, B., 1922, 67 A).—Samples of marri kino (*Eucalyptus calophylla*) contained tans 28·1—71·8%, soluble non-tans 4·8—11·1%, insoluble matter 8·6—20·1%, and water 10·0—54·5%. The colour was reddish and the p_H of the liquors 3·4—3·6. Autoclaving rendered a portion of marri kino insoluble at the expense of the tannin content. Autoclave treatment of marri kino with solutions of acid sodium sulphites at 110° for ½ hr. showed that 81% of the kino was solubilised with 8% of sodium metabisulphite, but the tans/non-tans ratio fell from 6·3 to 2·7 and good colour was obtained. Autoclaving at higher temperatures did not materially influence the composition. Autoclaving marri kino with normal sulphites yields more soluble matter but of darker colour. Similar experiments have shown that mixtures of from 1 : 3 to 1 : 6 normal and acid sodium sulphites are good solubilising agents on marri kino, and the colour is favourable. A marri kino extract prepared with 8% of a 1 : 4 mixture of normal and acid sodium sulphites had the following composition:—tans 61·5, soluble non-tans 26·8, insoluble matter 1·3, moisture 10·4, and ash 6·1%. Liquors prepared from this extract showed p_H 4·2 and the colour at 0·5% tannin content was 11·2 red units and 43·3 yellow units. It compares favourably with quebracho extract in respect of total solubles and tannin content, but contains too much insoluble matter. The tans/non-tans ratio is unfavourably low. Readily soluble products can be obtained by sulphiting marri kino at ordinary atmospheric pressures. D. WOODROFFE.

Fisetin; the cause of the fluorescein reaction with quebracho extract. Fluorescein and fluorescence reactions of quebracho, tizerah, mimosa, and urunday extracts. O. GERNGROSS and H. HÜBNER (Collegium, 1927, 426—431).—When quebracho, tizerah, mimosa, and urunday extracts are respectively fused with phthalic anhydride and zinc chloride, they show a strong fluorescein reaction, attributed to fisetin. Not only quebracho, tizerah, and mimosa extracts, but also urunday extract shows a strong yellow fluorescence when nitrocellulose (11% N) is dipped in 1 : 1000 solutions of these various extracts and examined in filtered ultra-violet light. This is due to fisetin. Ordinary cotton wool treated with a solution of urunday extract shows a lilac fluorescence in ultra-violet light owing to the presence of a lilac fluorescing substance not absorbed by the nitrocellulose. Fisetin is proved to be the cause of the fluorescein reaction with quebracho extract, since 1 mg. of fisetin gives the reaction whereas it fails with 1 mg. of quebracho extract, and, further, a sample of unsulphited liquid quebracho extract, from which the fisetin has been removed by extraction with ether, fails to show the reaction. The difficulty of removing the fisetin from quebracho extract indicates that it is present in a combined form. D. WOODROFFE.

Hide powder. W. R. ATKIN and F. C. THOMPSON (J. Soc. Leather Trades' Chem., 1927, **11**, 300—308).—Hide powder is collagen modified by liming which has opened up –CO·NH– linkages forming free carboxyl and amino-groups. The amphoteric properties of hide powder between p_H 2 and 9 are due to lysine, arginine, aspartic and glutamic acids. It is shown that the titration curve of B.12 hide powder corresponds to 26 free amino-groups; gelatin has 30. The isoelectric points of B.12 and gelatin are p_H 5·5 and 4·7, respectively. The isoelectric points of different hide powders can vary according to the numbers of free amino- and carboxyl groups present, which latter depend on the previous liming. The "opening up" of –CO·NH–

linkages has been measured for a series of hide powders by determining the number of c.c. of $0 \cdot 05N$-hydrochloric acid and $0 \cdot 05N$-caustic soda required when titrated between p_H 4·0 and 8·5. A comparison of these figures with those for the "relative specific surface" shows that the latter diminishes as the collagen molecule is more opened up. High relative specific surface figures correspond with low non-tannin results, which thus coincide with fewer free amino- and carboxyl groups. B.14 powder contains more free groups than B.13 and consequently requires more alkali per gram to bring it from p_H 6·66 to p_H 8·5. To obviate such differences, the use of unlimed hide from which the grain has been removed is suggested. Bennett's figures for the alkalinity of B.11 (cf. B., 1927, 52) are at variance with those of the Hide Powder Committee; the authors do not agree that the alkaline impurities are organic, and state also that the alkali does not remain in the powder and reduce non-tannin residue. D. WOODROFFE.

Determination of quebracho extract in other vegetable tanning extracts. O. GERNGROSS and H. HÜBNER (Collegium, 1927, 431—433).—The yellow fluorescence shown by cotton wool treated with solutions of quebracho extract and viewed in filtered ultra-violet light is visible at a dilution of 1 pt. per million. Solutions of the tanning extract under examination are prepared of different strengths, and the dilution at which the yellow quebracho fluorescence still appears is determined by the expression: % quebracho = dilution × $100/10^6$. By using a similar method of determination, 15—17% of fisetin was observed in a solid quebracho extract. D. WOODROFFE.

Determination of the activity of bating materials by Lenk's method. L. JABLONSKI and W. EGGERT (Collegium, 1927, 483—485).—2·5 g. of "cutrilin" and "oropon," respectively, were shaken with 200 c.c. of water, filtered, 10 c.c. of the filtrate added to 50 c.c. of a 10% solution of commercial (69·18%) gelatin, and kept for 1 hr. at 40°. The digestion was stopped by adding 10 c.c. of N-hydrochloric acid, the solution suitably diluted for gelatinisation, as determined by a preliminary test, and N-sodium hydroxide added to give p_H 7—7·5. The gelatin content was then determined by the time required for setting (cf. B., 1927, 86). The results, contrary to Lenk's observations, showed no difference between "cutrilin," a bacterial bate, and "oropon," a tryptic bate. The authors have compared the activities of these two bates respectively at different p_H values and for different periods of time, and the results differ from those obtained by Lenk (cf. B., 1927, 284). The Lenk method is merely a comparison of the proteolytic activities under the conditions of the experiment, and takes no account of any other properties. D. WOODROFFE.

Tanning materials of the British Empire (Bull. Imp. Inst., 1927, 25, 250—286).

PATENTS.

Process of tanning animal hides. CHEM. WERKE "HERKULES" G.M.B.H. (G.P. 439,551, 5.9.22).—The effluents of vegetable tanning processes are treated with alkalis or acid such as 5% sodium hydroxide, ammonia, sodium carbonate, bisulphite, or borax, respectively, filtered, diluted to contain 5—10% of dry residue, 2% of aniline is added, treated with 0·5% formaldehyde solution, and used for tanning hides, which it penetrates in 5—10 days. Alternatively, the hides may or may not be treated with a 2% aqueous solution of aniline oil, then given 2 days in a 0·5—1·0% solution of formaldehyde followed by 3 days in effluent liquors, which yields a white empty leather. D. WOODROFFE.

Process of tanning. PLAUSONS FORSCHUNGSINST. G.M.B.H. (G.P. 440,037, 31.1.24).—Ground products containing tannin, *e.g.*, quebracho wood, are ground in colloid mills or similar mechanism with water containing a dispersive agent, a material which speeds up the tannage or some product which improves the leather, and, without allowing the solids to settle out, the product is used as a tan liquor. Chromium and silica compounds, *e.g.*, kaolin, kieselguhr, or suitable oils, fats, or albumins, respectively, may be incorporated with the above mixture to produce modifications in the tanning properties. These various products reduce the time of tannage and produce a more homogeneous waterproof leather than can be obtained by the usual extract tannages (cf. E.P. 219,347; B., 1924, 841). D. WOODROFFE.

Process of tanning hides. S. SCHAPRINGER (G.P. 439,521, 16.7.25).—Hides are treated for 1—2 hrs. with dilute solutions of organic non-tans, *e.g.*, pyrocatechol, resorcinol, quinol, pyrogallol, phloroglucinol, protocatechuic acid, gallic acid, or weak tannins, *e.g.*, catechin, naphthols, quinones, kino tannin, cutch, and moringa, with or without the addition of tannin extracts, and then the tannage is completed in strong tannin extract liquors, *e.g.*, a mixture of chestnut, oakwood, and sulphited quebracho extracts (d 1·075) for 40—50 hrs., or a chestnut and mimosa bark extract liquor (d 1·057). This method is suitable for sole leather. D. WOODROFFE.

Process for tanning with metallic salts and salts of silicic acid. RÖHM & HAAS, A.-G. (E.P. 270,267, 7.4.27. Ger., 30.4.26).—Tanning liquors containing metallic salts (*e.g.*, chrome or potash alum) and salts of silicic acid are stabilised by adding acids or their salts of greater valency than two, *e.g.*, stannic acid, phosphoric acid, either alone or with uni- or bi-valent organic acids, *e.g.*, acetic, oxalic, or lactic acid. D. WOODROFFE.

Manufacture of a leather substitute. RESPRO, INC., and R. K. ABBOTT (E.P. 276,392, 7.4.26).—A reticulated, cement-impregnated sheet of unwoven fibres of loose or open texture is spread with one or more coats of a film-forming dressing, *e.g.*, nitrocellulose or soap–wax-shellac emulsion, containing a solvent which will act as a wetting agent towards the cement or will penetrate the sheet material and enclose the cement-coated fibres at and near the surface. The dressing of the required consistency may contain a liquid miscible with the cement in the sheet material and a colloidal material, *e.g.*, pigment. The surface-dressed sheet is subjected to pressure. Fibres impregnated with rubber cement may be similarly treated. D. WOODROFFE.

Manufacture of a translucent article. A. EGGIMAN and A. PEREA (U.S.P. 1,644,115, 4.10.27. Appl., 17.5.24. Fr., 12.6.23).—See E.P. 217,580; B., 1925, 683.

XVI.—AGRICULTURE.

Nitrification in soils. II. H. N. Batham (Soil Sci., 1927, 24, 187—203; cf. B., 1926, 70).—Nitrification tests in soil with monoaminodicarboxylic and diaminocarboxylic acids and with some heterocyclic compounds are reported. Arginine nitrifies more readily than aspartic or glutamic acid. Xanthine and uric acid are easily nitrified. Brucine and strychnine show very low nitrification values; nicotine is more readily nitrified than any of the other compounds tested, with the exception of ammonium sulphate. C. T. Gimingham.

Nitrate accumulation under a mulch. A. B. Beaumont, A. C. Sessions, and O. W. Kelly (Soil Sci., 1927, 24, 177—185).—In these experiments, the accumulation of nitrates was much greater in the soil of mulched plots than on that of adjoining cultivated plots. The mulch of waste hay and straw was applied twice yearly at the rate of 6—8 tons per acre for 4 years, and was left entirely undisturbed. Fruit trees were growing on the plots. The effect of the mulch on nitrate accumulation was the reverse of that obtained by Albrecht and Uhland (B., 1926, 25), but the conditions of mulching were different. C. T. Gimingham.

Difficultly soluble phosphates [in soil]. E. Ungerer (Z. Pflanz. Düng., 1927, A9, 321—345; cf. B., 1927, 55).—Continuing his studies on the solubility relationships of the tertiary phosphates of magnesium, calcium, aluminium, and iron, the author has investigated the influence of the presence of various compounds of uni- and bi-valent metals, and of certain adsorbents, such as clay and animal charcoal, on their solubility. Magnesium and calcium phosphates are stable in solutions of neutral or weakly alkaline reaction, iron and aluminium phosphates in acid solutions. Magnesium phosphate does not exist in the soil as such. The solubility of calcium phosphate is increased by small changes in reaction and by the presence of neutral salts, alkali and alkaline-earth carbonates, and adsorbents. On the other hand, most of the substances tried caused a reduction of solubility of iron and aluminium phosphates; only alkali carbonates and alkali-permutite caused an increase. The figures are discussed in relation to the results of vegetation experiments with oats.
C. T. Gimingham.

Effect of frost on the "availability" of phosphoric acid and potassium [in the soil]. B. Bätz (Z. Pflanz. Düng., 1927, A9, 346—363).—Neubauer's seedling method has been used to investigate the effect of freezing upon the available phosphoric acid and potassium in various types of soils. In the case of clay, loam, and chalk soils, freezing and thawing 20 times caused no significant increase of available nutrients; in a sandy soil there was a slight increase in the uptake of phosphoric acid by the plants; in a granite soil freezing brought about a definite increase in the amount of potassium taken up, but had no effect on phosphoric acid. The soil surface area, as measured by the hygroscopicity, is unaffected by freezing in clay, loam, and chalk soils, but an increase of area of 5·69% was observed in a sandy soil, and of 6·13% in a granite soil. It is considered that the increased availability of nutrients in the latter soils after freezing is due, not to any chemical effect, but to the increased surface area resulting in better utilisation by the plant roots.
C. T. Gimingham.

Relative tolerance of crop plants for aluminium. F. T. McLean and B. E. Gilbert (Soil Sci., 1927, 24, 163—175).—Different plants, grown in water culture, vary widely in their sensitiveness to the presence of soluble aluminium in the culture solution. With some crops (*e.g.*, barley, lettuce) growth is depressed by 2 pts. per million of aluminium, whereas with others (*e.g.*, maize, turnips) 14 pts. per million or more are required to show a toxic effect. The earliest evidence of injury due to aluminium is shown in the roots, and the aluminium appears to accumulate in the cortex. Precipitation of aluminium by phosphates was avoided by growing the plants for alternate half-weekly periods in a complete nutrient solution, and in a solution lacking phosphate but containing aluminium sulphate.
C. T. Gimingham.

Relation of soil reaction to active aluminium. A. W. Blair and A. L. Prince (Soil Sci., 1927, 24, 205—215).—Addition of acids or acid-forming substances to a normal soil (p_H 6·28) decreases the p_H and increases the amount of active aluminium, *i.e.*, that part of the aluminium of acid soils which produces a toxic effect on plants (cf. Burgess, B., 1923, 617 A). When basic materials or superphosphate were added to a strongly acid soil (p_H 4·07), there was an increase in p_H and a decrease in the amount of active aluminium. Lime and basic slag decreased the active aluminium to a greater extent than superphosphate. In general, the effects produced were greater the larger the additions of acid or basic materials. The results are in agreement with those obtained in field experiments where ammonium sulphate has been used for a period of years.
C. T. Gimingham.

Preparation of a soil for practical suspension analysis. A. von Nostitz (Proc. Internat. Soc. Soil Sci., 1926, 2, 305—308).—Thorough disintegration of the finer soil particles previous to mechanical analysis is achieved by drawing a rapid current of air through a suspension of the soil in a length of wide glass tubing.
A. G. Pollard.

Mechanical analysis of soils. I. M. Köhn (Z. Pflanz. Düng., 1927, A9, 364—375).—The proportion of "fine-sand" (diam. 0·02—0·2 mm. on the Atterberg scale) is of special importance in certain forest soils. The difficulties of determining this fraction accurately, either by settling and decantation or by means of sieves are pointed out. The pipette method and the processes of Wiegner and Odén are not concerned with the coarser fractions. A suitable apparatus for determining this fraction by an elutriation method is described.
C. T. Gimingham.

The Mitscherlich method [for determining the fertiliser requirement of soils]. Densch and Pfaff (Z. Pflanz. Dung., 1927, 6B, 385—398).—Numerous culture experiments are described, designed to test the constancy of Mitscherlich's values when applied to a wide variety of plants, soils, fertilisers, and external conditions of growth. It is concluded that whilst the method does not permit of such close control over soil fertility as is desirable, it is probably superior to

other methods for general application. The time and extreme care necessary for the successful operation of the method, together with the cost of materials, limits its application as a routine process. A. G. POLLARD.

Cellulose and its decomposition in the soil by micro-organisms. S. A. WAKSMAN (Proc. Internat. Soc. Soil Sci., 1926, **2**, 293—304).—In soils cellulose is decomposed mainly by fungi, and to a small extent by aerobic bacteria and actinomyces (cf. Waksman and Skinner, B., 1926, 959). Phycomycetes cannot decompose cellulose. Aerobic bacteria capable of decomposing cellulose are found in large numbers only in bog soils. Under aerobic conditions 50—65% of the carbon of cellulose is eliminated as carbon dioxide and 25—35% is utilised by the organisms concerned. The production of carbon dioxide under anaerobic conditions is much reduced and there is a corresponding increase in the proportion of intermediate substances (organic acids etc.). The amount of cellulose decomposed bears a definite proportion to the amount of nitrogen required by the organisms for cell formation. For fungi the ratio is 30 : 35, becoming wider for the usual mixed flora of the soil. Cellulose is not directly a parent substance of soil humus. Its contribution is limited to that portion of its carbon assimilated by the organisms, and which ultimately is decomposed with the production of humus. A. G. POLLARD.

Effect of protozoa and fungi on certain biochemical processes when inoculated into partially sterilised soil. C. E. SKINNER (Soil Sci., 1927, **24**, 149—161).—Reinoculation of soil, partially sterilised by heating, with one of the common soil amoebæ (*Hartmanella hyalina*), caused a reduction in the number of bacteria and a slight depression in the amount of carbon dioxide evolved and in the accumulation of ammoniacal nitrogen. Two species of soil fungi, similarly tested, caused a greater depression of ammonia accumulation than that due to the amoeba, and an increased evolution of carbon dioxide, which was greater than the depression caused by the amoeba. The action of fungi must be taken into account in attempting to explain the phenomena of partial sterilisation of soil. C. T. GIMINGHAM.

Fermentation characters of the root nodule bacteria of the leguminosæ. I. L. BALDWIN and E. B. FRED (Soil Sci., 1927, **24**, 217—230).—The relative powers of 60 cultures of root nodule bacteria to produce acid by fermentation of various sugars and other compounds have been studied. The characters thus obtained are sufficiently definite to separate the organisms into groups which correspond with the cross-inoculation groups. In some groups, *e.g.*, lucerne and clover strains, sub-divisions are found which correspond with those established by other workers by cultural and serological tests. C. T. GIMINGHAM.

Bacteriological methods for the investigation of soil fertility. H. R. CHRISTENSEN and H. L. JENSEN (Proc. Internat. Soc. Soil Sci., 1926, **2**, 309—323).—A classified summary of the trend of recent research in soil bacteriology. A. G. POLLARD.

Determination of the content of toxic substance in insecticides. III. Determination of polysulphide sulphur. J. BODNÁR and W. GERVAY (Z. anal. Chem., 1927, **71**, 446—458).—The polysulphide sulphur content of solutions of sodium or calcium polysulphide may be determined by the following method without the necessity of determining the thiosulphate sulphur. To 25 c.c. of $0\cdot1N$-silver nitrate solution is added with agitation a quantity of the polysulphide solution containing about 0·1 g. of excess sulphur. The precipitate of silver sulphide and sulphur liberated by the decomposition of the polysulphides is collected, washed with hot water until neutral, and digested under reflux for 30 min. with 20 c.c. of $0\cdot5N$-sodium hydroxide and 3 c.c. of perhydrol in 100 c.c. of water, whereby the free sulphur is converted into sodium sulphate, leaving the silver sulphide unaffected. The latter is collected on a filter and the excess of alkali titrated with sulphuric acid. The method is not applicable to barium polysulphide, the excess sulphur in which is determined preferably by Schulek's method (B., 1925, 241). The work of earlier investigators is critically reviewed, and comparative results obtained by various gravimetric and volumetric methods are tabulated. A. R. POWELL.

PATENTS.

Artificial drying of crops. B. J. OWEN (E.P. 275,015, 30.7.26. Addn. to E.P. 248,935; cf. B., 1926, 460).

Manufacture of phosphatic fertilisers. PRÉPARATION INDUSTRIELLE DES COMBUSTIBLES, and A. HOFFMANN (E.P. 269,199, 8.4.27. Fr., 8.4.26).—See F.P. 614,944; B., 1927, 663.

XVII.—SUGARS; STARCHES; GUMS.

PATENTS.

Extraction process and apparatus. W. F. HOLZHEUER (Re-issue 16,739, 20.9.27, of U.S.P. 1,587,646, 8.6.26).—See B., 1926, 718.

Molasses as a food (U.S.P. 1,643,666).—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Quinhydrone electrode method of hydrogen-ion concentration measurements. E. M. CROWTHER (J. Inst. Brewing, 1927, **33**, 459—463).—A description of the methods employed, with special reference to the liquids concerned in brewing operations. S. J. GREGG.

Fermentation test for silver compounds. TAYLOR.—See XX.

PATENTS.

Prevention of haze in pasteurised beer. HEINEKENS' BIERBROUWERIJ MAATSCHAPPIJ (Dutch P. 14,494, 12.2.23. Conv., 15.10.25).—The fermenting worts are treated with a saccharomycetes rich in proteolytic enzymes, *e.g.*, *Schizosaccharomyces Pombe* and *Octosporus*. C. RANKEN.

Manufacture of concentrated grape juice. SOC. ÉTABL. BARBET (F.P. 615,942, 29.9.25).—Fermentation of the juice in the grape is checked in the cellar by the addition of antiseptics, which at the same time furnish the small amount of sulphur dioxide necessary for the clarification of the must. After twice pressing as quickly as possible, the combined juices are continuously concentrated in a multiple-effect evaporator. C. RANKEN.

Sulphuring of wine or other liquids in bottles. SEITZ-WERKE G.M.B.H. (F.P. 617,210, 8.6.26).—A fixed amount of sulphur dioxide is mixed with a small quantity of wine or water, and an accurately measured portion of the mixture introduced into the bottles. C. RANKEN.

Manufacture of yeast. INTERNAT. YEAST CO., LTD., Assees. of W. H. F. BUHRIG (E.P. 252,193, 14.5.26. U.S., 14.5.25).—In the process, which is of the continuous addition–withdrawal type, propagation of the yeast is initiated with aeration in a dilute nutrient solution in a fermenter and relatively concentrated yeast nutrient solution added in excess of the attenuation of the yeast. After a period the liquid containing yeast is withdrawn, at a rate equal to the rate of inflow, into an auxiliary fermenter, where it is aerated and its concentration adjusted to that initially present in the main fermenter. When a sufficient amount of liquid has accumulated, the liquid containing yeast is withdrawn and the yeast separated. C. RANKEN.

Propagation of yeast. A. K. BALLS (U.S.P. 1,642,192, 18.7.23).—A culture of *Saccharomyces disjunctus* is added to and allowed to propagate in a mash of crude West Indian cane molasses. C. RANKEN.

Manufacture of yeast extract free from bitterness. VER. DES SPIRITUS-FABRIKANTEN IN DEUTSCHLAND (G.P. 441,457, 27.9.23).—By treatment of living or dried beer yeast with a 2—5% solution of hydrochloric acid at 35—80°, the yeast is hydrolysed and the bitter principles present are destroyed. C. RANKEN.

Rectification of ethyl alcohol. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (F.P. 614,913, 19.11.25. Belg., 20.11.24).—The impure alcohol is directly rectified whereby a decanted portion containing the impurities (methyl alcohol, acetone, etc.) is distilled in an auxiliary column, and the resulting gas mixture condensed or washed with water. C. RANKEN.

Removing gelatin from films (F.P. 613,074).—See XXI.

XIX.—FOODS.

Determination of sodium chloride in [preserved] yolk of egg. H. ULEX (Chem.-Ztg., 1927, **51**, 758—759).—Drying and igniting preserved liquid egg-yolk results in loss of chlorine even when sodium hydroxide is added before drying. Correct results may be obtained by boiling 5 g. of the yolk with 200 c.c. of water until coagulation ensues, diluting to 250 c.c., filtering off an aliquot part, evaporating to dryness with sodium carbonate and nitrate, igniting gently to destroy organic matter, dissolving the residue in water, and titrating with silver nitrate after acidifying with nitric acid. Alternatively, 4 g. of yolk are boiled for 2 min. in 100 c.c. of water, 50 c.c. of water, 20 c.c. of nitric acid (d 1·15), and 6 c.c. of 4% phosphotungstic acid are added in succession, the liquid is diluted to 200 c.c., 50 c.c. are filtered through a dry paper and treated with an excess of 0·1N-silver nitrate solution, and the excess silver is determined by titration with thiocyanate.

A. R. POWELL.

Loss of nutrients in the mechanical removal of water from potatoes. E. PAROW, A. STIRNUS, and W. EKHARD (Z. Spiritusind., 1927, **50**, 277—278).—The removal of water from potatoes by pressure depends on the preliminary treatment to which they are subjected, *e.g.*, fine or coarse grinding, freezing and subsequent thawing, cooking, mixing with crushed meal, or combinations of these. The amounts of proteins and salts removed at the same time are roughly proportional to the expressed water. The mechanical removal of water from potatoes results in a large loss of nutrients, that of the proteins being 30·5—49·7% and of the salts 39·5—67·2% of the amounts originally present.

F. R. ENNOS.

Sources of supply of vitamin-*A* and -*D*. O. ROSENHEIM and T. A. WEBSTER (Nature, 1927, **120**, 440).—Alternative sources of the vitamins present in butter are considered. It should be possible to produce commercially margarine containing a constant amount of vitamin-*A* and -*D* by the incorporation, respectively, of the liver fats of the sheep, calf, and ox (which are 200—1000 times as potent as butter) and of an extremely small proportion of irradiated ergosterol.

A. A. ELDRIDGE.

Comparison between the acids in acid foods as determined by Wiegner's method, and the hydrogen-ion concentration. K. NEHRING (Z. angew. Chem., 1927, **40**, 1058—1060).—The determination of the hydrogen-ion concentration of the aqueous extract is a good and rapid means of ascertaining the quality, and to a certain extent the composition of acid foods. Good quality foods have relatively low p_H values, and the acids present consist mainly of free lactic acid with a little acetic acid, combined acids and butyric acid being absent or present only in small amounts. Inferior varieties show higher values, the lactic acid is to a large extent replaced by butyric acid, and there is a substantial increase in the content of combined acids (acetic and butyric). For good and medium quality acid foods prepared by the cold process, the p_H values are less than 4·5 and between 4·5 and 4·8, respectively. When the p_H value exceeds 4·8 the food is bad and unfit for use. The p_H values observed are in good agreement with those calculated from the acids as determined by Wiegner's process, assuming the combined acids to be present as completely dissociated salts. F. R. ENNOS.

PATENTS.

Manufacture of bread. C. B. HILL and G. L. TINTNER, Assrs. to NORTHWESTERN YEAST CO. (U.S.P. 1,643,011—2, 20.9.27. Appl., [A] 10.9.25, [B] 26.10.25).—Nutrients for yeast, *e.g.*, sugar, malt extract, ammonium sulphate, calcium phosphate, and calcium sulphate, are mixed and kept for a sufficient length of time with (A) dry yeast in order to revivify it and thereby produce an active starter, which is compounded with a further predetermined portion of the yeast foods and used in the production of dough, or (B) with wet live yeast so that the yeast cells may multiply and become strengthened before being used in the dough in place of the usual dough requirements of the yeast, yeast foods, yeast activators, malt extract, and some of the sugar.

F. R. ENNOS.

[Gluten] food product. A. S. HOYT (U.S.P. 1,643,772, 27.9.27. Appl., 15.5.26).—Wet gluten gum, substantially freed from wheat oil and carbohydrates, including flour and starch, is cooked to a gruel and dried in thin, crisp flakes. F. R. ENNOS.

Manufacture of chocolate and other confectionery. D. McKINLAY (E.P. 276,860, 27.10.26).—In order to increase the vitamin content to a degree corresponding approximately with that of the ingredients in the raw state, manufactured confectionery is exposed to the action of ultra-violet rays by passing on an endless band under a quartz mercury-vapour lamp, the band being moved slowly or intermittently so as to present the confectionery to the action of the rays for a definite period, which can be varied according to the nature of the confectionery. F. R. ENNOS.

Process for artificially preparing natural mineral waters. O. WARBURG (E.P. 274,834, 22.6.27. Ger., 22.7.26. Cf. E.P. 259,612 ; B., 1927, 503).—In the manufacture of mineral waters which contain magnesium and calcium salts as well as alkali bicarbonates, sulphates, and free carbonic acid, the magnesium, mixed with the alkali bicarbonate, is added to water in the form of non-hygroscopic magnesium potassium chloride, made by heating the commercial salt to 130—140°, and the calcium, together with the alkali bisulphate, as calcium sulphate finely ground in a colloid mill.
F. R. ENNOS.

Process and product of treating molasses. J. H. LEFTWICH (U.S.P. 1,643,666, 27.9.27. Appl., 31.5.24).—Low-grade or final by-product molasses is mixed with finely-divided decolorising carbon to improve the tinctorial characteristics, and the mixture is used as a stock food ingredient. F. R. ENNOS.

Manufacture of butter. S. KARPINSKY and J. S. ANDERSON (U.S.P. 1,644,254, 4.10.27. Appl., 16.1.24. Fr., 21.11.23).—See E.P. 214,567 ; B., 1924, 533.

Method of preparing foods. C. BIRDSEYE, Assr. to GEN. SEAFOODS CORP. (Re-issue 16,740, 20.9.27, of U.S.P. 1,608,832, 30.11.26).—See E.P. 257,222 ; B., 1927, 457.

Electric furnace [for baking bread etc.]. ÉTABL. SABLYET (E.P. 257,237, 22.7.26. Belg., 22.8.25).

Reducing, grinding, or refining apparatus for chocolate, cocoa, etc. F. G. FRYER (E.P. 277,177, 11.6.26).

XX.—ORGANIC PRODUCTS ; MEDICINAL SUBSTANCES ; ESSENTIAL OILS.

Determination of water in mixtures of benzene and alcohol. D. PETERS (Z. angew. Chem., 1927, **40**, 1011—1013).—On adding an equal volume of light petroleum to a mixture of benzene and alcohol the mixture separates into two layers at a definite temperature, which depends on the water content of the alcohol used in making the mixture and on the benzene content of the mixture. The temperature of separation has been plotted against the benzene content for mixtures of benzene with alcohol samples of varying water content ; in every case the curve is a straight line and the lines corresponding with varying contents of water are parallel to one another, so that, if the benzene content of a mixture is known, and the temperature of separation when mixed with an equal volume of petroleum is ascertained, the water content of the mixture may be found by reference to the graph. The benzene content of an unknown mixture is determined by shaking the liquid with water and measuring the separated benzene ; to a further 2 c.c. of the mixture are added 2 c.c. of petroleum, and the liquid is cooled slowly with stirring until separation into two layers occurs. In case separation occurs immediately on mixing, the liquid is warmed gently until it becomes homogeneous, then allowed to cool slowly until separation occurs. A. R. POWELL.

Determination of gallic acid in tannin-free gallic acid. M. HIRSCH (Chem.-Ztg., 1927, **51**, 718—719).—A boiling aqueous solution containing 0·2 g. of the substance is precipitated with 10 c.c. of an acetic acid solution of bismuth nitrate (15 g. of bismuth nitrate and 30 g. of glacial acetic acid diluted with water to 250 c.c.). After remaining a short while on the steam bath, the precipitate is collected on a Gooch filter, washed with hot water till free from acid, then with alcohol and with ether, and dried at 45—50°. The precipitate of bismuth subgallate, $C_6H_2(OH)_3CO_2Bi(OH)_2$, contains 45·79% of crystallised gallic acid.
F. R. ENNOS.

Application of the U.S.P. X. yeast fermentation test to colloidal silver compounds. H. M. TAYLOR (J. Amer. Pharm. Assoc., 1927, **16**, 820—824).—The yeast test, *i.e.*, the inhibitory action of the silver compound upon a yeast-sucrose solution, has been tried on ten commercial silver preparations. It is useful only in differentiating between "mild" and "strong" samples and measures only the silver ion concentration, which is not directly related to the germicidal value of the preparation. The simpler and quicker method of Keelan (*ibid.*, 1926, **15**, 277) is recommended in place of the yeast test for distinguishing between such preparations. The germicidal values of the above samples, as determined by the Squibb test, show that some preparations are extremely active against *Staphylococcus aureus* after 1 min. E. H. SHARPLES.

Optical identification of alkaloids. G. L. KEENAN (J. Amer. Pharm. Assoc., 1927, **16**, 837—839).—The procedure used for the determination of the refractive indices of alkaloids by the immersion method is described, together with the method for the detection and differentiation of atropine, heroin, morphine, cocaine hydrochloride, and the sulphates of brucine, morphine, codeine, and quinine (cf. Wherry, A., 1918, ii, 339).
E. H. SHARPLES.

Occurrence of a number of varieties of *Eucalyptus dives* as determined by chemical analyses of the essential oils. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. N.S. Wales, 1927, **61**, 54—67).—From observations on the compositions of the essential oils from trees of this species it is concluded that, in addition to the species type, three varieties exist. These are indistinguishable morphologically, but yield distinctive oils. The type oil contains piperitone 40—50%, phellandrene 40%. Oils from the three varieties contain, respectively, piperitone 5—15%, phellandrene 60—80%, and piperitol ; piperitone 10—20%, cineole 25—50%, with some phellandrene ; and cineole 45—75%, piperitone under 5%, phellandrene absent or trace only.
S. I. LEVY.

Volatile oil of *Hypericum perforatum*. E. R. MILLER (J. Amer. Pharm. Assoc., 1927, **16**, 824—

828).—Steam-distillation of the whole plant gives an average yield of 0·1146% of oil having d_{15}^{25} 0·8126, α_D −16·43°, n_D^{20} 1·45903, and containing α-pinene and probably one or more sesquiterpenes. A fraction (b.p. 140—142°, d_{20}^{20} 0·7135, $n_D^{18.5}$ 1·4048) probably consists of an aliphatic hydrocarbon not hitherto known to occur in an essential oil. E. H. Sharples.

Monardella oil. E. R. Miller (J. Amer. Pharm. Assoc. 1927, **16**, 828).—Steam-distillation of the air-dried whole plant of *Monardella lanceolata*, Gray (Western pennyroyal), yields 1% of oil having d_{15}^{25} 0·9392, n_D^{18} 1·4908, α_D + 17·4°, and probably consisting principally of pulegone. E. H. Sharples.

Colour reactions of the ethereal oils and of some of their constituents. L. Ekkert (Pharm. Zentr., 1927, **68**, 577—583, 593—602).—The colour reactions of furfuraldehyde, sucrose, salicylaldehyde, vanillin, cinnamaldehyde, and piperonal with a very large number of the essential oils, and with many of the commoner constituents of the oils, in alcoholic solution and in the presence of varying quantities of concentrated sulphuric acid, are tabulated. S. I. Levy.

***p*-Aminophenol.** Caesar.—See XI.

Fumigant. Cotton and Roark.—See XXIII.

Patents.

Dissolving insoluble substances [*e.g.*, camphor]. Ges. für Physik.-Chem. Untersuchungen (Swiss P., 117,768, 5.2.25).—Camphor is dissolved in a mixture of alcohol and oleic acid in such proportions that the solvent has no deleterious action on the specific properties of the camphor. A. R. Powell.

Preparation of methyl formate. M. Mugdan and J. Wimmer, Assrs. to Consortium für Elektrochem. Ind. (U.S.P. 1,642,689, 20.9.27. Appl., 22.1.26. Ger., 22.1.25).—Formaldehyde vapour is passed over finely-divided copper at 100—250°. H. Holmes.

Preparation of ethyl hydrogen sulphate. Comp. de Bethune (E.P. 273,263, 26.5.27. Fr., 24.6.26).—Ethylene is passed through a series of absorption vessels, the later vessels being maintained at higher temperatures, each vessel being supplied independently with fresh sulphuric acid at such a rate as to keep the concentration of ethyl hydrogen sulphate in the vessel at its optimum concentration for absorption purposes (cf. E.P. 221,512; B., 1925, 299). The outflowing solutions of ethyl hydrogen sulphate in sulphuric acid are mixed together and worked up for alcohol in the usual way. The preliminary absorption at the lower temperature removes substances which would decompose at the higher temperatures. C. Hollins.

Synthesising and separating higher alcohols. G. Patart (E.P. 250,563, 29.3.26. Fr., 9.4.25).—In the synthesis of higher alcohols by repeated passage of carbon monoxide and hydrogen under pressure over a heated catalyst, the reacted gases are first cooled under pressure to condense alcohols etc., and carbon dioxide is then removed by further cooling under pressure before returning the remaining gases to the heating tubes and reaction chambers. The cooling may be assisted or effected by partial expansion, and the liquefied carbon dioxide is used to cool uncondensed gases. The catalysts are alkali or alkaline-earth chromates, manganates, molybdates, tungstates, uranates, vanadates, etc., mixed with an equivalent or less of a catalyst favouring the production of methyl alcohol, *e.g.*, a mixture of 83 pts. of zinc oxide and 200 pts. of potassium chromate, or 10·5 pts. of manganese dioxide and 200 pts. of barium tungstate. The gas mixture, preferably water-gas containing 48 vols. of hydrogen to 42 vols. of carbon monoxide, which corresponds with the equation $14CO + 16H_2 = 2C_5H_{11}\cdot OH + 4CO_2 + 4H_2O$, passes at high pressure directly, or through a heat exchanger, to the catalyst at 320—400°, and thence through a series of four condensers cooled respectively by gas returning to the reaction vessels, by alcohols to be evaporated, by cold water, and by cooled gases. The uncondensed gas mixture, freed thus from water and alcohols, passes to a second series of condensers cooled respectively by cooled gases, by evaporated carbon dioxide, by brine at −20° to −35°, and by the liquid carbon dioxide evaporating at a determined lower pressure. The carbon dioxide in the mixture is thus liquefied, and cooled uncondensed gases are returned by way of the condenser jackets to the circuit. The reaction product collected below the first series of condensers is freed from dissolved gases by partial release of pressure to 1—2 atm., the liberated gases being scrubbed in brine and returned to the compressor. The higher alcohols are salted out from the aqueous product and evaporated in the jacket of the second condenser of the first series, the vapours passing to a rectifying plant. A suitable plant for carrying out all these operations is illustrated. C. Hollins.

Manufacture of vinyl esters [acetate]. F. W. Skirrow and O. W. Herzberg, Assrs. to Canadian Electro Products Co., Ltd. (U.S.P. 1,638,713, 9.8.27. Appl., 8.11.20).—Glacial acetic acid is treated with acetylene in presence of mercuric sulphate at 80°, and the vinyl acetate and ethylidene diacetate formed are separated by fractional condensation. The diacetate is heated with sulphur dioxide at 150° to give a further yield of vinyl acetate. T. S. Wheeler.

Manufacture of a polyolefine glycol ether. J. G. Davidson, Assr. to Carbide & Carbon Chemicals Corp. (U.S.P. 1,633,927, 28.6.27. Appl., 20.7.25).—Olefine oxides react with alcohols or with glycol monoethers under pressure at 70—250° to give polyolefine glycol monoethers, *e.g.*, β-*hydroxy*-β′-*ethoxyethyl ether*, b.p. 190°, is formed by the condensation of ethyl alcohol with ethylene oxide (2 mols.). These compounds are of value as solvents for cellulose esters (cf. B., 1926, 714). T. S. Wheeler.

Substituting hydroxyl groups for the acid radicals in liquid esters of inorganic acids or for halogens in liquid derivatives of hydrocarbons. [Preparation of alcohols by hydrolysis of inorganic esters, organic halides, etc.] E. E. Ayres, jun., and E. E. Haabestad (E.P. 241,889, 14.10.25. U.S., 21.10.24).—Inorganic esters, organic halides, mercaptans, etc. which are liquid at the temperature of reaction, are heated with an alkali and either an organic ester of the same alcohol or a sodium salt of an organic acid. If an

organic ester is used it is hydrolysed by the alkali, giving the desired alcohol and the sodium salt. The sodium salt is stated to react with the inorganic ester forming the organic ester, which is readily hydrolysed. This cycle of reactions is applied to the hydrolysis, *e.g.*, of chlorinated pentane by aqueous sodium hydroxide in presence of amyl oleate or sodium oleate (or common soap). The soap facilitates reaction by emulsifying the mixture. The process is carried out advantageously under pressure (*e.g.*, in a closed vessel at 150°), and the amyl alcohol is distilled off, after removal of sodium chloride solution, as the pressure is gradually released. C. HOLLINS.

Converting difficultly soluble or insoluble colloidal carbohydrate ethers into new soluble products. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 277,111, 10.6.26).—Carbohydrate ethers, particularly cellulose ethers, are converted into soluble modifications by treatment with a depolymerising agent, such as mineral acid, or zinc chloride and hydrochloric acid, with or without addition of swelling agents or solvents. Cellulose ethyl ether, when treated with a mixture of alcohol (3 vols.), water (3 vols.), and concentrated sulphuric acid (1·5 vols.), becomes readily soluble in benzene or benzene-alcohol, the solutions on evaporation giving films equal to those from known soluble cellulose ethers. Glacial acetic acid containing 0·1% of sulphuric acid has a similar effect. C. HOLLINS.

Manufacture of nuclear aralkylated [naphthyl] alkyl ethers and their sulphonic acids [wetting-out agents]. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 277,098, 8.6.26).—β-Naphthyl *iso*amyl ether is heated at 140—160° with benzyl chloride and a metal catalyst (iron filings) until no more hydrogen chloride is evolved. The cooled mixture is sulphonated, giving an amorphous, soap-like product with excellent wetting-out properties. Other naphthyl ethers and other aralkyl halides (*e.g.*, methylbenzyl chlorides) may be used. C. HOLLINS.

Manufacture of salts of 2-phenylquinoline-4-carboxylic acid. M. L. CROSSLEY and M. L. DOLT, Assrs. to CALCO CHEMICAL Co. (U.S.P. 1,638,832—4, 16.8.27. Appl., 9.6.25).—(A) *Lithium* 2-*phenylquinoline*-4-*carboxylate,* from aqueous lithium carbonate solution and the acid, crystallises with 6·5 and 1 mol. of water. (B) *Mercuric* 2-*phenylquinoline*-4-*carboxylate* and (C) *ferric* 2-*phenylquinoline*-4-*carboxylate* are prepared from the sodium salt and soluble mercuric or ferric salts. T. S. WHEELER.

Manufacture of derivatives of amino-metal-mercaptosulphonic acids and salts thereof. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 270,729, 4.5.27. Ger., 5.5.26).—Soluble sulphomethyl derivatives are obtained by the action of sodium formaldehyde-bisulphite on amino-metalmercaptosulphonates. Compounds obtained from sodium β-amino-α-aurothiolethane-α-sulphonate and 4-amino-2-argentothiolbenzene-1-sulphonate are described. C. HOLLINS.

Manufacture of condensation products of aldehydes with ketones. I. G. FARBENIND. A.-G. (E.P. 264,830, 17.1.27. Ger., 19.1.26).—The alkaline condensation of aldehydes with ketones is conducted in the absence of water, solid alkali or an alkali-metal compound of a β-keto-alcohol being used. From acetaldehyde and acetone in presence of the potassium compound of diacetone-alcohol (methyl β-hydroxy*iso*butyl ketone), methyl β-hydroxypropyl ketone ("hydracetylacetone") is obtained; from propaldehyde, acetone, and solid potassium hydroxide, methyl β-hydroxy-*n*-butyl ketone, b.p. 80—85°/10 mm.; and from butaldehyde, methyl ethyl ketone, and solid potassium hydroxide, ethyl β-hydroxy-*n*-amyl ketone, b.p. 95—96°/10 mm. In similar manner are obtained $\Delta^{\alpha\gamma}$-pentadienyl methyl ketone (crotylideneacetone), phenyl propenyl ketone (b.p. 120—125°/10 mm.), cinnamenyl methyl ketone (b.p. 155°/12 mm.), and 2-ethylidene*cyclo*hexanone. C. HOLLINS.

Sedative and hypnotic ureides [carbamides]. F. BOEDECKER (U.S.P. 1,633,392, 21.6.27. Appl., 9.9.26. Ger., 3.3.23).—α-Alkylacylcarbamides, in which one at least of the alkyl radicals is unsaturated, are of value as sedatives and hypnotics. *E.g.*, α-*iso*propyl-Δ^{γ}-pentenoyl chloride (*iso*propylallylacetyl chloride) readily condenses with carbamide to α-iso*propyl*-Δ^{γ}-*pentenoylcarbamide*, m.p. 191°, the same product being obtained from α-*iso*propyl-Δ^{γ}-pentenoamide and cyanic acid, heated in carbon tetrachloride at 100° under pressure. Ethyl *sec.*-butylmalonate when condensed with allyl bromide in presence of sodium ethoxide gives *ethyl* sec.-*butylallylmalonate*, b.p. 145—148°/12 mm., which is hydrolysed to the corresponding *acid*, and when heated at 190° yields the monocarboxylic acid derivative, β-*methyl*-α-*allylvaleric acid*, b.p. 228—230°. This compound is treated with thionyl chloride at 100° to form β-*methyl*-α-*allylvaleryl chloride*, b.p. 60°/12 mm., which with carbamide gives β-*methyl*-α-*allylvalerylcarbamide*, m.p. 147—148.° T. S. WHEELER.

Production of methane. F. KLATTE and J. SÖLL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,643,663, 27.9.27. Appl., 30.11.25. Ger., 3.12.24).—See E.P. 244,076; B., 1927, 316.

Production of new ester mixtures. W. CLAASEN (U.S.P. 1,643,619, 27.9.27. Appl., 17.3.26. Ger., 14.4.25).—See E.P. 250,910; B., 1926, 769.

Manufacture of *dl*-nerolidol. L. RUZICKA, Assr. to M. NAEF & Co. (U.S.P. 1,644,546, 4.10.27. Appl., 19.3.24. Switz., 22.3.23).—See E.P. 213,250; B., 1925, 378.

Manufacture of calcium salts of inositolphosphoric acid. A. GAMS and M. GIRARD, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,644,246, 4.10.27. Appl., 1.12.25. U.K., 29.3.23).—See E.P. 218,014; B., 1924, 731.

Manufacture of solutions containing organic phosphorus and complex gold compounds. L. CASSELLA & Co., LTD. (E.P. 253,946, 21.6.26. Ger., 22.6.25).—See U.S.P. 1,635,169; B., 1927, 797.

Benzoxazolonearsine oxides [arsenoxides]. L. BENDA and O. SIEVERS, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,635,167, 12.7.27. Appl., 19.6.25. Ger., 25.6.24).—See E.P. 257,361; B., 1926, 932.

Benzoxazolonearsinic acids. L. BENDA and O. SIEVERS, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,635,168, 12.7.27. Appl., 27.7.25. Ger., 11.8.24).—See E.P. 261,133; B., 1927, 125.

Preparation of salts of alkaloids and acetylaminohydroxyphenylarsenic acid [acetamidohydroxyphenylarsinates of quinine and quinidine]. F. Billon, Assr. to Etabl. Poulenc Frères (U.S.P. 1,643,692, 27.9.27. Appl., 19.3.26. Fr., 28.3.25).—See E.P. 249,849 ; B., 1926, 769.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Production of photographic images on cellulose acetate film. A. J. Hall and R. A. Hill (J. Soc. Dyers and Col., 1927, **43**, 291—292).—Cellulose acetate dyed with S.R.A. Blue III, IV, V, VII, Violet II, and Heliotrope I suffers a change of shade when subjected to the action of nitrous acid as used for the development of indigosol dyes (cf. Friedländer, B., 1926, 705), the original shade being restored by exposure to direct sunlight, but not to ordinary electric light. The restoration of colour is sufficiently rapid in the case of S.R.A. Blue III to allow the production of photographic images on cellulose acetate materials. *E.g.*, Celastoid (cellulose acetate film containing plasticising agents) dyed with S.R.A. Blue III in a 1·5% soap solution at 85° so that it also becomes partially delustred and opaque, is immersed for 10—15 min. in a 10% solution of sodium nitrite and then in 7% sulphuric acid without previous rinsing whereby it becomes pale reddish-yellow. Subsequently it is exposed for 45 min. behind a photographic negative to light from a Fadeometer, and a clear blue print thus obtained. Such prints are much more permanent than an ordinary unfixed print on P.O.P., but become uniformly blue in colour on prolonged exposure to daylight. Similar results were obtained on Celastoid dyed with Thionol Sky Blue PX, this being bleached to white by means of sodium hyposulphite instead of nitrous acid, but the restoration of colour by exposure to sunlight is extremely rapid, and even occurs slowly in the dark. In all cases the most satisfactory results were obtained on cellulose acetate materials which had been previously delustred by treatment with a hot solution of soap. A. J. Hall.

Fog formation by dyestuffs. Lüppo-Cramer (Z. wiss. Phot., 1927, **24**, 408—415).—The position with regard to chemical fogging brought about by the presence of sensitising or desensitising dyestuffs in the gelatin–silver halide emulsion is reviewed. The composition of the developer and the nature of the dye are both important factors in determining whether fogging will or will not occur. The rôle of silver bromide–reducing agent adsorption complexes is discussed. R. A. Morton.

Theory and practice of the Carbro process. C. Lighton (Phot. J., 1927, **67**, 362—373, 409—421).—Detailed theoretical and practical consideration is given to the function of each of the reagents in the Carbro baths. The importance of following the diffusion of the various reagents is emphasised. The flattening of Carbro prints with increase in the concentration of dichromate is mainly due to the fact that in the case of dilute dichromate the ferrocyanide has to diffuse a comparatively long way into the tissue before it is all oxidised, whilst with concentrated dichromate the corresponding diffusion is shorter, and the hardening action is confined to a comparatively thin layer. Chromic acid has a similar but much more vigorous action than dichromate. The function of acid in the Carbro bath is to liberate chromic acid from the dichromate. The difficulty of satisfactorily reproducing the high-lights in Carbro is due to the varying dilution obtained with hand squeegeeing, and a mechanical squeegee is described which gave greater consistency in the rendering of high-lights. The use of a hardening agent such as formaldehyde is both unnecessary and undesirable. The only useful function of the second bath (hydrochloric and acetic acids, and formaldehyde) is to counteract the unequal rate at which the ferricyanide bleaches the different parts of the silver image. The most satisfactory baths have the following composition :—First bath, chromic acid 0·04%, potassium dichromate 0·16%, potassium ferricyanide 2·0%, potassium bromide 2·0% ; second bath, chromic acid 0·04%, potassium dichromate 0·16%. The gradation curves of Carbros made under various conditions are discussed theoretically. W. Clark.

Patents.

Manufacture of photographic silver halide emulsions. I. G. Farbenind. A.-G., Assees. of A.-G. für Anilin-Fabr. (E.P. 258,237, 14.8.26. Ger., 14.9.25).—In a silver halide emulsion are incorporated decomposition products of animal or vegetable proteins, prepared by subjecting the protein to alkaline or enzymic hydrolysis together with partial oxidation. The course of hydrolysis and oxidation is observed by studying the change in refractive index, or by making test emulsions. The products may be added during the manufacture of the emulsion, or to one or more of the constituents of the emulsion, or to the prepared emulsion. High-speed emulsions may in this way be obtained from inactive gelatins. W. Clark.

Removal of gelatin from photographic films etc. A. C. Roerich and E. Moreau (F.P. 613,074, 7.7.25).—A bacterium, probably of the proteus group, found in putrid waters, is cultivated in the usual media in presence of gradually increasing quantities of silver salts. The culture is distributed in a very dilute aqueous solution containing ferrous sulphate, ammonium sulphate, sodium silicate, magnesium sulphate, sodium sulphate, potassium sulphate, sodium nitrate, calcium chloride, and sodium chloride, and the support to be degelatinised is treated with the mixture at 36°. The gelatin is liquefied in ½—3 hrs., and after sterilisation with formalin the support can be used again. W. Clark.

Blue-printing. E. H. Gay (F.P. 608,621, 3.4.25).—Blue-print or similar paper is treated with a solution of a salt, preferably ferric chloride, which liberates iodine from its salts, and containing also starch, gelatin, and tartaric acid. On exposure beneath a drawing, the unexposed parts retain their reactivity, and when moistened with a solution of potassium iodide, the drawing appears in blue-black lines. W. Clark.

Decoration of fabrics by the action of light. M. Michels (G.P. 441,150, 3.3.25. Cf. Swiss P. 109,722 ; B., 1926, 77).—Ferrocyanide or metal oxides forming a mordant are produced by reaction with the silver resulting from exposure of sensitive silver salts, and are

then dyed with basic or mordant dyes. *E.g.*, cotton fabrics are soaked in a solution of ferric ammonium citrate and silver nitrate, dried, exposed under a negative, washed with thiosulphate solution and water, bathed in a solution of potassium ferricyanide, rinsed, treated with copper sulphate solution, and then dyed with Thioviolet. W. CLARK.

Pictures by the transfer process. R. VON ARX (U.S.P. 1,643,529, 27.9.27. Appl., 17.12.25. Ger., 24.12.24).—See E.P. 245,163; B., 1926, 997.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Explosive. A. C. SCOTT and MEXCO, LTD. (E.P. 276,715, 4.5.26 and 3.3.27).—Alkali salts with readily available oxygen are mixed with similar ammonium salts in presence of enough moisture to cause partial dissolution of one or both salts with subsequent formation of a joint mass. Ammonium nitrate and potassium chlorate are suitable salts. A solid fuel may be introduced into the mixture before interaction, or a liquid fuel, in which explosive nitro-compounds may be present, may be added after pulverisation of the joint mass. The latter may be pulverised to pass 16-mesh/in. but not 120-mesh/in. The space for fuel between the crystals may be reduced by pressure to a volume not exceeding 40% of the total volume, including voids, occupied by the crystals. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Salinity of the Lake Washington Ship Canal. E. V. SMITH and T. G. THOMPSON (Univ. Washington Eng. Exp. Sta., 1927, Bull. 41, 33—41).—*Sampling*:—In one method of sampling a marine pump with about 50 ft. of $\frac{3}{4}$-in. garden hose graduated in feet was utilised, but sampling with this apparatus was tedious. A second device comprised a reversing water-bottle (Eckman type) attached to a wire cable graduated in feet, which, when lowered to the desired depth, was caused (by means of a brass messenger and a spring) to make a half-turn to a position where it automatically enclosed a sample of water. The two methods give practically identical results. *Analysis*:—Halides, as chlorides, were determined by Mohr's method, the titrations with silver nitrate in the presence of potassium chromate being carried out in yellow light. Hydrogen sulphide when present was first removed by boiling. Neutralisation of acid solutions with alkali using Methyl Orange as indicator and then proceeding with the silver nitrate titration gave good results. The Mohr method is applicable to solutions within the range p_H 4·2 to that of sea water. For determination of the halides as chlorides in sea water the Mohr method is as accurate as the gravimetric process or the Volhard volumetric process as modified by Ditmar. The presence of the carbonate ion in sea water has no effect upon the Mohr determination. The phosphate ion is present in such minute quantities that it is insufficient to produce precipitation. W. T. LOCKETT.

River water control. J. ZINK and F. HOLLANDT (Z. angew. Chem., 1927, **40**, 1062—1064).—The partial cleansing of rivers from magnesium chloride pollution probably occurs through substitution of calcium for magnesium by reaction with the zeolite-bearing clays of the river bed. Treatment of magnesium chloride solutions with clays and mixtures of clay and sand shows displacement of small quantities of magnesia from solution up to 2430 mg. for every 1 kg. originally present, with dissolution of an equivalent amount of lime. Repeated passage of the solution through the material is more effective than shaking them together and keeping for 24 hrs. F. R. ENNOS.

Testing of water contaminated with coal gas. REGENSTEIN (Chem.-Ztg., 1927, **51**, 737—738).—Minute quantities of phenol are detected by Millon's reagent, the water being treated with sodium chloride and tartaric acid, and the phenol then extracted in ether. Carbon monoxide, acetylene, and ethylene are detected by the reduction of permanganate solution or by reaction with chlorine water, whilst carbon monoxide is determined by absorption in palladium chloride or by the blood test, and acetylene by treatment with ammoniacal cuprous chloride. The usual tests are applied for hydrocyanic acid and thiocyanates. H. D. GREENWOOD.

Dichloroethane-carbon tetrachloride mixture; a new non-burnable, non-explosive fumigant. R. T. COTTON and R. C. ROARK (J. Econ. Entomology, 1927, **20**, 636—639).—A mixture of 3 pts. by vol. of *s*-dichloroethane and 1 pt. by vol. of carbon tetrachloride is very effective against stored-product pests, *e.g.*, clothes moth, furniture beetle, black carpet beetle, etc.; it is about 5 times as toxic as carbon tetrachloride, and is well adapted for general fumigation work in air-tight vaults. Tests indicate that it would be equally effective against the flour beetle, rice weevil, Indian meal moth, and the saw-tooth grain beetle. The mixture has no corrosive action on metals nor any bleaching or staining action on textiles of any sort, and when vaporised in a fumigating chamber is non-injurious to fabrics. Unless breathed in high concentrations and for a protracted period of time it is not dangerous to human life. For general fumigation work in gas-tight chambers a dosage of 14 lb. per 1000 cub. ft. with a 24-hr. exposure at a temperature of 18° is recommended; the fumigant is applied by pouring it into a shallow pan near the ceiling of the vault. W. T. LOCKETT.

Ferric aluminium sulphate. NEGRI and BADO.—See VII.

PATENTS.

Varying the circulation of sludge in the purification of sewage. M. W. MILLS, J. F. BOLTON, J. BOLTON, and R. AMES (E.P. 276,483, 12.7.26).—In an activated sludge system, to vary at different times the quantity of sludge returned from the settling tanks to the aerating tank and thus vary the amount of sludge in circulation, a simple type of centrifugal pump, so constructed as to be variable as to capacity and easily regulated, is used to lift the sludge from the settling tanks. The pump comprises essentially a rotary device having vanes or blades rotating in a horizontal plane and an adjustable disc whereby the depth of the passage-ways in the rotary impeller may be varied according to the amount of sludge required to be pumped.
W. T. LOCKETT.

Zeolites (U.S.P. 1,642,880). **Base-exchange silicates** (E.P. 277,082).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOVEMBER 25, 1927.

I.—GENERAL; PLANT; MACHINERY.

Soft rubber filter-press plates and frames. H. E. Fritz and J. H. Clark, jun. (Ind. Eng. Chem., 1927, **19**, 1151).—Plates and frames of semi-hard rubber were tested in the filtration of corrosive liquids at 65 lb./in.^{2} and gave satisfactory results with the following: hydrochloric acid, caustic soda, ammonia solution, copper sulphate, etc. (all at any concentration), and sulphuric acid (up to 50%). The limit of temperature is, however, 40—65° according to the material undergoing filtration. No trouble from leakage or distortion occurred. Hard rubber, though resistant, is too fragile and liable to cold-flow for use in this connexion.

C. Irwin.

Stainless iron in chemical plant construction. Mitchell.—See X.

Patents.

Hardness testing device. C. H. Wilson (E.P. 277,517, 15.10.26).—A test point is caused to penetrate the test piece under a definite load. The point and piece are caused to approach under a minor load until they engage, this motion being effected independently of the size of the test piece. A major load is then applied, causing the penetration, and is kept on long enough to take the reading. The point is then withdrawn ready for another test. All the above motions are performed automatically. B. M. Venables.

Apparatus for analysing substances by means of Röntgen or cathode rays. F. Dessauer. Assce. of Metallbank & Metallurgische Ges. A.-G. (E.P. 253,090, 18.5.26. Ger., 5.6.25. Addn. to E.P. 252,207; B., 1927, 735).—In apparatus constructed in accordance with the prior patent, the primary Röntgen or cathode rays are caused to fall obliquely upon, or to brush against, the substance to be analysed, so that the electronic emission of the characteristic secondary radiation exceeds that of the non-characteristic radiation.

J. S. G. Thomas.

Solidification of liquid substances. I. G. Farbenind. A.-G., Assecs. of K. Rast (G.P. 442,358, 12.12.24). —Liquids, *e.g.*, methyl alcohol, castor oil, liquid hydrocyanic acid, are converted into solids by treatment with magnesium ethoxide in the presence of small quantities of water. B. Fullman.

Filters. H. A. Vallez (E.P. 277,546, 1.2.27).—The filter is of the type in which a number of leaves are rotated within a casing, slowly when cake building and rapidly when discharging, the filtrate being withdrawn through a hollow shaft. The filter leaves are arranged radially and longitudinally, and besides being attached directly to the shaft they are supported by transverse discs. B. M. Venables.

Bag filter with outflow pipes disposed on one side for filter bags closed on all sides. W. H. Scheidt (F. Scheibler) (E.P. 272,209, 30.5.27. Ger., 7.6.26).—A method of connecting the outflow pipes to the external pipes is described. B. M. Venables.

Controlling the saturation temperature of gases and apparatus therefor. Carrier Engineering Co., Ltd., and S. L. Groom (E.P. 276,221, 11.1.27).—The gas is passed through a chamber containing sprays of water the temperature of which is controlled by a thermostat fixed in the gas stream. Various methods are described: (*a*) the water from the sprays is cooled by a liquid, *e.g.*, brine, the flow of which is regulated; (*b*) the water is cooled in an evaporator, the evaporation of the refrigerating liquid being controlled by the flow of condensing water; (*c*) a regulated stream of cold water is added to the circulating water in a tank prior to the chamber and any excess water is withdrawn by a system of weirs; (*d*) the addition of cold water is made through a three-way valve controlled by the thermostat.

A. C. Monkhouse.

Optical pyrometer. G. Keinath, Assr. to Siemens & Halske A.-G. (U.S.P. 1,644,340, 4.10.27. Appl., 23.11.25. Ger., 29.5.25).—See E.P. 244,691; B., 1926, 114.

Working fluid for refrigeration. A. A. Kucher (U.S.P. 1,645,198, 11.10.27. Appl., 27.4.27).—See E.P. 208,547; B., 1924, 621.

Apparatus for ascertaining the composition of gaseous mixtures. H. Heinicke, Assr. to P. Jung (U.S.P. 1,646,247, 18.10.27. Appl., 16.5.24. Ger., 29.11.23).—See E.P. 225,549; B., 1925, 696.

[Automatic system for] furnace regulation. Brit. Thomson-Houston Co., Ltd., Assees. of A. R. Smith (E.P. 264,852, 20.1.27. U.S., 20.1.26).

[Economical firing of boiler] furnaces. B. Ljungström (E.P. 277,396, 16.3.26).

Machines for discharging and charging furnaces or retorts. Gibbons Brothers, Ltd., and N. G. Cook (E.P. 278,081, 30.6.26).

[Sealing device for elements of] heat-exchange apparatus. Serck Radiators, Ltd. From Zimmermann & Co., Ludwigshafen a./Rhein Maschinen- & Apparatebau (E.P. 277,556, 16.2.27).

Distributing cock for use in subjecting solid material to the action of fluids. L. Mourgeon (E.P. 277,726, 21.5.26).

Gas-fired furnaces (E.P. 263,817).—See II.

Volumetric gas-displacement apparatus (E.P. 277,869).—See XXIII.

II.—FUEL ; GAS ; DESTRUCTIVE DISTILLATION ; MINERAL OILS.

Free-burning carbonised fuels for the open fire. H. H. Greger (J. Akita Min. Coll., 1927, No. 3, 52 pp.).—The theory of combustion in the open grate is discussed, and in connexion therewith an adsorption theory is suggested based on the assumption of fields of force in the micropores of the charcoal or other fuel similar to the magnetic field of force in a solenoid. The suitability of a smokeless fuel for burning in the open grate is dependent on other factors than the ignition temperature, such as, for example, the activation due to oxidation during the initial period of burning ; it has been investigated by studying the combustion of a layer of the fuel about 1 cm. in depth, contained in an iron tube 3·55 cm. in diameter, in a regulated downward current of air. The fuel (10—20-mesh) rests on a layer of quartz sand (20—30-mesh), and is ignited by an electrically-heated nichrome spiral lying on the surface. The spiral is heated for 3 or 5 min. and a constant air current passed until the resulting combustion has ceased. The results show the existence of a critical rate of passage of air at which there is a sudden rise, from about 10 to about 80%, in the amount of fuel burnt. This, the " combustion threshold," is the minimum rate which will maintain combustion in the fuel layer. The series of fuels, " black " oak-charcoal, lignite semi-coke, " white " oak-charcoal, semi-coke from a bituminous coal, and a commercial charcoal show a progressive increase in the " combustion threshold " rate from 0·6 to 1·5 litres/min. No continued combustion is maintained with anthracite or high-temperature coke under the experimental conditions. The influence of various factors, *e.g.*, particle size, water content, activation, preheating, on the value of the critical rate has been investigated. A simple apparatus for the rapid determination of the ignition temperature has been designed, in which the fuel is heated by the passage through it of a current of air which passes first through an outer hot layer of sand.

A. B. Manning.

Gaseous fuels for furnace heating. R. Wiggington (J. Soc. Glass Tech., 1927, **11**, 293—297).—In producer practice a moderate amount of steam in the blast improved the heating value of the gas, but a further increase was disadvantageous. Sufficient steam was necessary to prevent clinker trouble, but a gas rich in carbon monoxide was preferable to one rich in hydrogen, since it gave a longer flame and greater radiating power, and was less liable to surface combustion. Control of blast saturation temperature was essential, for which purpose a positive blower was better than the usual steam jet.

A. Cousen.

Differentiation of the conceptions coal, lignite, and peat. G. Stadnikov and N. Proskurnina (Brennstoff-Chem., 1927, **8**, 305—306 ; cf. B., 1926, 729).—Lignite exhibits a much greater power of taking up ferric oxide from a solution of ferric chloride than coal of the same particle size. The extent of surface adsorption is probably about the same for both materials, but the humic constituents of lignite exert a chemical attraction for ferric oxide not shown by coal.

W. T. K. Braunholtz.

Relation between the physical nature and adsorptive capacity of coal samples. D. J. W. Kreulen (Brennstoff-Chem., 1927, **8**, 306).—It is emphasised in criticism of the work of Stadnikov and Proskurnina (cf. previous abstract) that the adsorptive capacity of a coal sample can be exactly defined only by relating it to unit area of adsorbing surface. It is also essential that only fresh coal samples are used, since oxidised coal will show similarities to lignite.

W. T. K. Braunholtz.

Formation of hydrocarbons from lignite coke at 500°. F. Fischer and H. Pichler (Brennstoff-Chem., 1927, **8**, 307—309).—When steam is passed over activated lignite semi-coke, prepared by carbonising lignite briquettes at 500° and activating by heating the product with a mixture of steam and air, in the presence of a metallic catalyst (*e.g.*, carbonates of manganese, cobalt, or iron, or manganese dioxide) traces of methane and of naphthalene are obtained. The former is, however, produced by the direct action of heat on the semi-coke and disappears on prolonged heating. Contrary to the results obtained by Hofmann and Groll (B., 1927, 239), no higher hydrocarbons are found in the gas. If steam is replaced by dry hydrogen, traces of hydrogenation products (*e.g.*, ethane) are formed.

W. T. K. Braunholtz.

Gas industry : past, present, and future. W. J. A. Butterfield (J.S.C.I., 1927, **46**, 399—407 t).—An address reviewing progress of public gas supply in Great Britain from 1812 onwards, and contrasting it with that of other public utility services. Official testings under the provisions of the Gas Regulation Act, 1920, covered, at the end of 1926, 71·5% of the gas supplied. They showed that throughout the general strike and coal stoppage of 1926 71·17% of the gas tested was not below its normal calorific value, and a further 18·76% was not as much as 5% below. Comparatively few and unimportant gas undertakings only have been found since 1922 to have supplied in any quarter gas of lower average calorific value than that on which their charge by the " therm " is based. The mean deficiency for all such cases is 1·25%, and in no quarter was more than 0·075% of the gas sold found to be deficient—apart from exceptional cases in the coal emergency period of 1926. Gas supplies now range from 200 to 600 B.Th.U. per cub. ft. in calorific value, but 90% of the whole fall between 440 and 560 B.Th.U. There are at least three gas works sending gas under relatively high pressure to rural districts 20 miles distant. Sales of gas in Great Britain in 1926 were more than four times as great as in 1882, and the consumption registered by each consumer's meter averaged 32,900 cub. ft. in 1926 as compared with 33,360 cub. ft. in 1882. On the basis of heat equivalents, the sales of gas increased between 1922 and 1925 at 2½ times the rate of increase in the sales of electricity by statutory undertakings.

Disposal of liquor effluents from gas works. Inst. Gas Eng., Res. Comm. Rep. **I.** A. Parker. **II.** A. C. Monkhouse (Gas J., 1927, **179**, 101—104).—I. The principal effluent from gas works arises from the manufacture of ammonium sulphate. This effluent, which contains phenols, thiocyanate, and thiosulphate, interferes with the self-purification of rivers into which

it is discharged and increases the difficulties of sewage purification when discharged into sewers. Of the 45—90 gals. of effluent produced per ton of coal carbonised, 90—85% forms the spent liquor discharged from the ammonia stills and 10—15% the "devil liquor" from the heat-exchangers which follow the sulphuric acid saturator in which the ammonia is absorbed. The oxygen absorption capacity for effluent liquors ranges from 300 to 1000 pts./10^5, whereas the corresponding figure for average domestic sewage is 10 pts./10^5. The volume of effluent liquor to be treated is usually 0·5—1·0% of the volume of sewage, and in this proportion it can be successfully treated if sudden changes in the proportion of effluent to sewage are avoided. Purification of the effluent by bacterial filtration reduces its oxygen absorptive capacity by 95%, but the initial cost of the process is large. Partial purification by the use of dephenolating towers reduces the oxygen absorptive capacity by only 20—30%. Admixture with sewage appears to be the only satisfactory method at present known, and it is suggested that arrangements should be made to run the effluent into sewers in regulated quantity.

II. Experiments made at Hinckley Gas Works relating to the problem are described. The total volume of effluent, of which 14% was "devil liquor," was 1·4 times the volume of ammoniacal liquor produced. The oxygen absorption capacity of the "devil liquor" was 20% of that of the total effluent. The circulation of liquor from a common tar and liquor well through the hydraulic main is unsatisfactory in that it produces a loss of ammonia and the formation of thiosulphate and thiocyanate. The storage of liquor and tar in a common well favours the dissolution by the liquor of higher tar acids, which should be avoided by early tar and liquor circulation. Preliminary experiments on the purification of effluent liquor by biological filtration have shown that with a rate of flow of 60 gals. per day per cub. yard of filtering material in each of two filters, 85% purification can be obtained with a mixture containing 8% of effluent liquor. Filter beds matured in this manner can be used for the treatment of effluent liquor diluted with bacterially purified effluent. S. PEXTON.

Cholesterol as parent of petroleum. N. D. ZELINSKI (Ber., 1927, **60**, 1793—1800).—A mixture of cholesterol and aluminium chloride is cautiously heated in a small flask with a free flame; the weight of the distillate is 66% of that of the cholesterol, and includes gaseous products condensed at —70°. The liquid distillate is treated with steam and the volatile portions are then distilled, yielding mainly a fraction, b.p. 60—150°, d_4^{20} 0·7416, n_D^{22} 1·4154, which is somewhat unsaturated. After hydrogenation in the presence of palladised asbestos at 170°, a saturated product, b.p. 60—150°, n_D^{21} 1·4136, is obtained consisting mainly of cyclic compounds. From it a mixture of hexane and heptane hydrocarbons, 1 : 4-dimethyl*cyclo*hexane, and methyl*cyclo*hexane are derived. The less volatile oils have b.p. 150—400°/atmos. pressure without decomposition. In composition they closely resemble several natural petroleums containing oxygen. With increasing b.p. the fluorescence of the individual fractions increases in intensity and the magnitude of the dextro-rotation increases, corresponding to that of fractions of Campina and Schodnica petroleum. Treatment of the fractions of cholesterol oil with sulphuric acid causes diminution of the density of those of higher b.p., renders them almost inactive optically, and removes the fluorescence; precisely similar observations are made with natural petroleums. The possible presence of traces of unchanged cholesterol in the higher fractions cannot be examined by the usual colorimetric tests. The carbonised residue which remains in the distillation flask yields a resin poor in hydrogen when extracted with benzene. Oleic, stearic, and palmitic acids are converted by aluminium chloride into mixtures of liquid and solid hydrocarbons which differ in character from those derived from cholesterol. H. WREN.

Esthonian oil shale: origin of oil shales. P. N. KOGERMAN (Sitzungsber. Naturforsch.-Ges. Univ. Tartu, 1927, 34, 166—182; cf. B., 1927, 354).—Esthonian oil shale, "kukersite," contains approximately 43·4% of organic matter (kerogen). Analysis of the latter corresponds nearly to an empirical formula $(C_6H_8O)_n$. Kerogen from Scottish shale oil has an empirical formula $(C_6H_{10}O)_n$; hence the kerogen of kukersite is less saturated. Some derivatives of hydroaromatic compounds, *e.g.*, resins, unsaturated aldehydes, etc., have the same general formula as the kerogen of kukersite. The kerogen is insoluble in all ordinary organic solvents, and is apparently a highly polymerised "resinous substance," very resistant to the action of reagents (*e.g.*, fusion with potash or sulphur) and depolymerised by the action of halogens and heat only. The character of the distillates obtained on thermal decomposition of the shale recalls the character of those obtained from resins and rubber.
R. C. ODAMS.

Fluorescence of low-temperature distillation products of oil shales. M. WITTLICH (Brennstoff-Chem., 1927, 8, 309—310).—Oils obtained from the low-temperature distillation of different oil shales exhibit, when examined by means of a quartz lamp, different fluorescence according to their origin and method of production and purification. The fractions can be arranged in order of their b.p. by means of their different fluorescence. W. T. K. BRAUNHOLTZ.

PATENTS.

Drying process for fuel. A. V. LIPINSKI (Swiss P. 117,203, 22.10.25).—Fuel oil is completely burnt in a suitable combustion chamber, the products are mixed with several times their volume of air, and this gas mixture is passed into the drying chamber. In this manner the full heating value of the fuel is utilised, and the drying gases are free from sulphur dioxide, carbon monoxide, and other objectionable constituents.
A. B. MANNING.

Apparatus for washing and treating coal. ILLINGWORTH CARBONIZATION CO., LTD., and S. R. ILLINGWORTH (E.P. 276,382, 22.2.26).—Coal is treated with a liquid such as trichloroethylene in washing troughs through which run conveyors arranged to give an up-and-down motion to the coal. The clean coal passes to a pitch tank and from there to a dryer. The dirt is also dried and the vapours from the two dryers pass to a condenser, the water being separated and the liquid returned to

the tanks feeding the troughs. Any vapour left in the gas is recovered in a tower packed with charcoal.
A. C. MONKHOUSE.

[Coal] grinding and drying apparatus. J. E. BELL, Assr. to COMBUSTION ENGINEERING CORP. (U.S.P. 1,641,409, 6.9.27. Appl., 12.1.22).—The grinding mill is placed in a conduit through which a heating medium is passed.
T. S. WHEELER.

Treatment of fuels. L. B. DEVILLARS (Swiss P. 117,304, 23.10.25).—Coal, coke, or lignite, etc. is powdered, dried *in vacuo*, and mixed with a fluid material which increases its calorific value and which solidifies in the cold. A suitable material consists of 20—60% of tar, 3—20% of magnesite, 0·5—15% of naphthalene, and 10—35% of chalk. The treated fuel is dried and given a coating of waterproof material.
A. B. MANNING.

Cleaning of carbonaceous materials. R. LESSING (E.P. 276,723, 27.5.26).—After the removal of fine dust the coal is fed into the middle of a bath of liquid of suitable density, *e.g.*, an alkaline solution of calcium chloride. The liquid is stirred at the point of entry of the coal, and the "coal" and "dirt" are separated. The two fractions are removed to separate towers and the solution removed by downward displacement with a continuous flow of weak bath solution and then water. A definite line of demarcation between the liquids is obtained, and the solution is returned for further use, or, if necessary, first concentrated. The loss of solution is reduced to less than 1% of the weight of coal.
A. C. MONKHOUSE.

Preparation of humin substances from coal. M. PIETTRE (F.P. 582,400, 6.9.23).—Powdered coal is treated with concentrated nitric acid; the mixture is cooled during addition of the acid, is then warmed slowly to 70°, and maintained at that temperature for 60—80 hrs. The washed and dried nitrated product, which has an average nitrogen content of 4·65%, is dissolved in concentrated alkali and reduced by treatment with an excess of powdered zinc or iron. Reduction requires several days, and is complete when, on acidifying and drying the product, this no longer burns with the evolution of nitrous fumes. The washed and dried product is black and lustrous, has a conchoidal fracture, and possesses chemical properties similar to those of the humic acids. Both nitrated and reduced products are soluble in pyridine.
A. B. MANNING.

Coke ovens. A. W. MATHYS. From TAR & PETROLEUM PROCESS CO. (E.P. 277,256, 11.5.27).—A rectangular coke oven is heated by combustion flues under the sole of the coking chamber, and extending the entire length of the latter. Regenerators arranged beneath the flues supply preheated air alternately to each end of each flue, where also means for supplying gas are provided. Each combustion flue is provided with movable tunnel nozzles for distributing a portion of the air supply from the regenerators and for discharging waste-heat gases from the flue to other regenerators; these permit control of the uniformity of heating in the flues.
A. B. MANNING.

Coke oven. W. H. WRIGHT, Assr. to FOUNDATION OVEN CORP. (U.S.P. 1,643,532, 27.9.27. Appl., 7.6.21).—A coke oven has coking chambers which are wider at one side than at the other. The combustion flues in the flue wall are arranged in groups, each of which communicates independently of the others with a pair of sole flues, which have a single opening at the end, to be used alternately for exhaustion of combustion gases and, on reversal, for admission of air. The effective areas of the passages from the sole flues to the combustion flues and the latter also are graduated in size continuously, increasing from one side to the other of the coking chamber as the width of the latter increases.
A. B. MANNING.

Manufacture of hard coke. P. DVORKOVITZ (E.P. 276,181, 15.10.26. Addn. to E.P. 249,901; B., 1926, 477).—An alkali, *e.g.*, lime or soda, is mixed with the coal or other material to be carbonised before or during the preliminary heating prior to carbonisation.
A. C. MONKHOUSE.

Solid smokeless fuel. ILLINOIS ANTHRACITE CORP., Assees. of C. S. LOMAX and W. M. GRANT (Can. P. 263,017, 19.10.25).—A bed of carbonaceous material of constant depth is maintained in a generator, the combustion in which is of such a nature that a relatively small proportion of the volatile matter, but no solid material, is consumed. The material in the generator is stirred continuously.
A. B. MANNING.

Dry-cooling of gas-works coke. P. GYGAX, and A.-G. KESSELSCHMIEDE RICHTERSWIL (Swiss P. 117,957, 13.11.25).—The coke is discharged into a jacketed container, the jacket of which is filled with some suitable liquid and is connected with a heat exchanger.
A. B. MANNING.

Cooling granular combustible material such as coke, semi-coke, etc. DEMAG A.-G. (G.P. 442,353, 26.6.24).—Separation into the various grain sizes and cooling of the separated material take place simultaneously during the course of conveyance, and the treated material is then partly or wholly remixed, also during its conveyance. The procedure economises in time and cost.
A. B. MANNING.

Motor fuel. G. MORETTI (F.P. 617,481, 12.6.26. Italy, 16.6. and 30.6.25).—A mixture of alcohol with a small quantity of acetone, hydrogen peroxide, potassium chlorate, resin, and grease is saturated with acetylene.
A. B. MANNING.

Motor fuel. H. P. BASSETT (U.S.P. 1,641,520, 6.9.27. Appl., 26.7.22).—Aniline (0·2%) and nitrotoluene (0·1%) are added to a hydrocarbon motor fuel to reduce the tendency to knock.
T. S. WHEELER.

Pulverulent fuels for use in operating internal-combustion engines. I. G. FARBENIND. A.-G. (E.P. 270,703, 2.5.27. Ger., 7.5.26).—To prevent the abrasive action of the mineral constituents the fuels are treated with hydrofluoric acid to remove wholly or partly the silicic acid.
A. C. MONKHOUSE.

Apparatus for the purification of graphite by froth flotation. A. GALLOIS (F.P. 617,697, 13.11.26).—A mixture of graphite with a special liquid is conveyed automatically from a mixer into frothing apparatus.
L. A. COLES.

Manufacture of carbon granules for telephone transmitters. F. O. BARRALET (E.P. 277,129, 17.6.26).

—A pure organic compound, *e.g.*, furfuraldehyde, sugar, is heated with a small quantity of a carbonising agent, *e.g.*, sulphuric acid, and the colloidal carbon so produced is subjected to further heat treatment, *in vacuo*, under pressure, or in a non-oxidising atmosphere. The dense product thus obtained is then crushed and graded.

A. B. Manning.

Production of hard homogeneous combustible material or objects such as picture frames, blackboards, lighting structures, etc. from peat, peat moss, lignite, and similar substances. B. Jirotka (E.P. 276,471, 30.3.26).—The material to be treated is first broken up by mechanical or chemical means and, after compression by a worm-feed, passes between rotating discs where it is subjected to a rubbing action and water is removed. The product obtained dries readily or can be moulded when plastic.

A. C. Monkhouse.

Gas-fired furnaces, more particularly for coke- and gas-producing furnaces. C. Still (E.P. 263,817, 23.12.26. Ger., 28.12.25).—In a vertical-flued coke oven of the regenerative type two regenerators are arranged for each half oven. When poor gas is used as the heating gas the gas passes through one of the regenerators to a separate horizontal flue, and thence to the burner nozzles, while the other regenerator is used for preheating the air. When rich gas is being used, by an adjustment of dampers both regenerators are used for the preheating of air, the regenerators being connected to the same horizontal flues. This principle is applicable to furnaces where recuperative or regenerative devices are employed. A. C. Monkhouse.

Production of enriched illuminating or power gas from coal or other fuel capable of being distilled. J. W. Gibson (E.P. 276,065, 17.5.26).—In a water-gas plant the carburettor is dispensed with by mixing the oil for enrichment with the steam supply to the generator. The mixture passes through a superheater fixed in the generator wall before admission to the fuel bed. A. C. Monkhouse.

Manufacture of water-gas. Humphreys & Glasgow, Ltd., and J. C. Stelfox (E.P. 276,753, 4.6.26).—In a complete gasification plant in which the fuel in the retort is heated internally by the hot gases from the generator below, the formation of a central core of partially carbonised fuel is prevented by suspending a core of metal or refractory material down the centre of the retort. A. C. Monkhouse.

Generation of carburetted water-gas from bituminous fuel. O. Misch (E.P. 276,530, 8.11.26).—A retort which is superimposed on a generator is heated externally by burning water-gas under pressure in a jacket surrounding the retort. The temperature of the lower end of the retort is maintained at 700° or higher. The gases then pass in a spiral direction round the retort and are used for generating steam. The temperature of the upper part of the retort is regulated by a water jacket. Tar or oil for carburetting is admitted in a ring-shaped pipe half-way down the retort.

A. C. Monkhouse.

Manufacture of gas from heavy oils. C. Chilowsky E.P. 271,907, 19.5.26. Belg., 16.7.27).—The heavy oil is atomised by preheated air and steam, and the temperature raised to 900—1000° by partial combustion of the oil. The mixture is passed over an incandescent refractory or metallic catalyst, when a nearly complete transformation is effected into non-condensable gas and very light hydrocarbons. R. C. Odams.

Treatment of gases by the employment of solid catalytic and other reactive agents. D. M. Henshaw, S. G. Watson, and W. C. Holmes & Co. (E.P. 276,736, 12.6.26).—An apparatus for the continuous purification of gases (*e.g.*, removal of hydrogen sulphide by iron oxide, moisture or benzol vapours by silica gel, etc.) consists of an elongated casing with foraminous walls containing the purifying agent and situated inside a chamber to which the gases are admitted. Baffles to cause the gases to pass repeatedly through the purifying agent by a zig-zag path and means for renewing the agent as desired without discontinuing the process are provided. C. O. Harvey.

Elimination of hydrogen sulphide from gases. C. Still (E.P. 271,805, 4.8.26. Belg., 25.5.26).—In a process for the removal of hydrogen sulphide from gases by washing with a suspension of ferrous and ferric hydroxides or carbonates, and regeneration of the latter by oxidation with air, the iron content of the suspension is maintained at not more than 2%, and the sulphur-containing foam from the regenerator is floated off into another vessel. After separation from the excess washing liquid, the sulphur is recovered from the foam by centrifuging. The regenerator consists of a tall narrow tower filled with the liquid, the oxidising air being introduced under pressure at the bottom. The wash liquid may be made alkaline, either by absorption of ammonia from the gas or by addition of alkali; the liquid is then circulated until a highly concentrated solution of thiosulphate is formed. A. B. Manning.

Distillation of carbonaceous materials. H. Nielsen and B. Laing (E.P. 276,407, 6.3.26).—Carbonisation takes place in two stages in an internally-heated rotary retort. In the first stage (185—580°) the products of distillation flow in the same direction as the inert heating gas, thus preventing the cracking of the oils and deposition of graphitic carbon on the pores of the coke; in the second stage (580—1200°) superheated water- or producer-gas is used as the heating gas. The coke obtained contains 1—4% of volatile matter, and is easily ignited. The oil obtained has a static coefficient of friction of 0·100—0·185 at 12—18°, and is suitable for lubricating purposes. A. C. Monkhouse.

Low-temperature distillation. Bamag-Meguin A.-G., and O. Heller (E.P. 268,745, 11.3.27. Ger., 1.4.26).—The material after passing through a dryer is fed into one of two rotary drums containing previously-heated refractory material made of tiles in lattice formation of varying designs. Carbonisation is effected at 450—850° and the distillation gases are withdrawn in an opposite direction to the moving charge. When one drum is in use the other is heated, and the heating gases pass subsequently to the dryer and then to a dust extractor, where the fuel recovered is used as pulverised fuel in the furnace. A. C. Monkhouse.

Low-temperature distillation of carbonaceous material. SYNTHETIC AMMONIA & NITRATES, LTD., R. E. SLADE, and C. F. R. HARRISON (E.P. 276,522, 13.10.26).—The finely-divided material is preheated to 100—200° and fed into a horizontal rotary retort through which steam superheated to 450° is passed in counter-current flow. Very little permanent gas is produced, and the products of distillation are recovered without condensation of steam by injecting a small quantity of water and then washing with oil at 140—150°. The steam is returned to the superheater for re-use. A portion of the circulating gas is removed from time to time as the permanent gas accumulates. The system may be worked under increased or diminished pressure and catalysts mixed with the fuel. A. C. MONKHOUSE.

Cracking of mineral oil and other hydrocarbon material. J. F. P. DE LA RIBOISIERE (E.P. 276,532, 12.11. and 31.12.26).—Liquid or solid fuels are cracked under pressure, *e.g.*, 5—10 kg./cm.^{2}, and the vapours produced are passed through a tower or column packed with Raschig rings or some highly porous material. The tower is maintained at a constant temperature by the introduction into the gas stream of a liquid, *e.g.*, water or benzol, under pressure. A. C. MONKHOUSE.

Process of carrying out chemical reactions with liquids [cracking of hydrocarbons]. E. H. LESLIE and B. R. TUNISON (U.S.P. 1,644,736, 11.10.27. Appl., 4.9.20).—A supply of liquid hydrocarbons is uniformly and intermittently distributed over the surface of a second supply of liquid hydrocarbons, the temperature of the latter being higher than that of the former, so that a conversion temperature is attained at least as high as the b.p. of one of the oil fractions. R. C. ODAMS.

Conversion [cracking] of oil. W. M. CROSS, Assr. to GASOLINE PRODUCTS CO., INC. (U.S.P. 1,644,991, 11.10.27. Appl., 3.4.23).—Oil is cracked in the liquid phase, and the products are then vaporised and fractionally condensed, a regulated liquid level being maintained in the latter stages. Selected portions of the condensate are returned, together with the charging stock, to the heating and condensing stages. R. C. ODAMS.

Preparation and recovery of light oil or motor fuel from crude mineral or shale oil, tar oils, or carbonaceous materials. E. SCHULTZ (E.P. 277,042, 9.3.26).—A modification of the process previously described (E.P. 254,011 ; B., 1926, 779) wherein oils or carbonaceous materials are heated in a retort, and the vapours are passed (at temperatures below that of the retort) through a converter containing salt, lime, zinc chloride, etc., consists in treating the heavier and lighter fractions separately and employing for the conversion substances such as montmorillonite, marialite, bentonite, lime and metallic sodium, aluminium chloride, or mixtures of these substances. C. O. HARVEY.

Production of liquid hydrocarbons and derivatives thereof from coal, tar, etc. I. G. FARBENIND. A.-G. (E.P. 277,273, 13.3.26. Addn. to E.P. 247,217, and 249,155 ; B., 1927, 601, 805).—In the conversion of coal, tars, mineral oils, etc. by treatment under pressure and at elevated temperatures with gases containing hydrogen or oxides of carbon, the cold as well as the hot parts of the apparatus are made of a metal which does not react with carbon monoxide. Other reducing gases may be used. C. O. HARVEY.

Transforming methane into a carburetting agent similar to petroleum. H. SPINDLER, Assee. of A. GOUDET (E.P. 255,493, 19.7.26. Fr., 18.7.25).—Methane, either alone or with other gases capable of combining with hydrogen, is subjected to high pressure (70—200 atm.) and temperature (500—950°), when partial decomposition takes place with formation of hydrogen. The gaseous mixture is cooled to 250—350° and passed over a catalyst consisting of a mixture of zinc chloride, active carbon, aluminium and magnesium powders, and iron scale previously treated with alkali hydroxides. Liquefiable hydrocarbons of a higher order than methane are produced. R. C. ODAMS.

Apparatus for the manufacture of carbonised fuel. S. R. ILLINGWORTH, Assr. to ILLINGWORTH CARBONIZATION CO., LTD. (U.S.P. 1,645,861, 18.10.27. Appl., 27.9.24. U.K., 29.10.23).—See E.P. 223,624 ; B., 1925, 3.

Manufacture of active carbon. J. N. A. SAUER (U.S.P. 1,641,053, 30.8.27. Appl., 29.7.24. U.K., 9.8.23).—See E.P. 206,862 ; B., 1924, 548.

Production of carbon [black]. C. MATLOCK, Assr. to MONROE-LOUISIANA CARBON CO. (Re-issue 16,765, 11.10.27, of U.S.P. 1,458,351, 12.6.23).—See B., 1923, 820 A.

Process of refining mineral oils. T. HELLTHALER, Assr. to H. STINNES-RIEBECK MONTAN- U. ÖLWERKE A.-G. (U.S.P. 1,645,530, 18.10.27. Appl., 1.8.25. Ger., 3.12.24).—See G.P. 421,858 ; B., 1926, 263.

Manufacture of liquid fuels. M. MÜLLER-CUNRADI and W. WILKE, Assrs. to BADISCHE ANILIN- & SODA-FABR. (U.S.P. 1,646,014, 18.10.27. Appl., 15.7.24. Ger., 14.1.24).—See E.P. 226,731 ; B., 1925, 163.

Apparatus for discharging coke ovens etc. STETTINER CHAMOTTE-FABR. A.-G. (E.P. 276,325, 8.8.27. Ger., 17.8.26).

Doors for gas retorts. R. HADDAN. From STETTINER CHAMOTTE A.-G. VORM. DIDIER (E.P. 278,269, 9.4.27).

III.—TAR AND TAR PRODUCTS.

PATENTS.

Manufacture of organic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 277,394, 13.3.26).—Aromatic hydrocarbons or phenols (or tar fractions containing these), when passed with excess of steam over hydrating and dehydrogenating catalysts or catalyst mixtures at 300—500°, are converted into lower homologues. *o*-Cresol over copper on bauxite gives phenol ; sulphur-free naphthalene, or crude naphthalene containing phenols, over nickel and alumina on china clay gives benzene, toluene, *o*-xylene, etc. Nickel, cobalt, iron, and copper favour dealkylation, the first three being also suitable for removing fused nuclei. Catalyst poisons should be removed from the vapours. Oxygenated compounds may first be reduced with hydrogen to hydrocarbons. The side-chains are removed chiefly as carbon dioxide and hydrogen, with

a little methane and ethylene; the gases produced are thus suitable for further catalytic reactions, *e.g.*, the production of alcohol or of pure hydrogen.
C. HOLLINS.

Light oil from tar oils (E.P. 277,042 and 277,273).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

PATENTS.

Manufacture of emulsions. I. G. FARBENIND. A.-G. (E.P. 267,534, 10.3.27. Ger., 10.3.26).—Aryl dihydrogen phosphates, diaryl hydrogen phosphates, or their salts are used alone or with other emulsifiers for the production of emulsions of non-phenolic organic compounds in liquids in which the compounds are substantially insoluble. Examples are sodium ditolyl phosphate ("sodium dicresyl phosphate") for emulsification of *cyclo*hexanone in water, oleic acid in water, aniline in water, and *cyclo*hexanone and benzene in water; sodium ditolyl phosphate, disodium tolyl phosphate or sodium dinaphthyl phosphate for dispersion of Indanthrene Blue RS in water; sodium diphenyl or dixylyl phosphate for dispersion of Indanthrene Golden Orange G in water.
C. HOLLINS.

Production of quinone derivatives [leuco-esters of vat dyes]. B. WYLAM, J. E. G. HARRIS, H. A. E. DRESCHER, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 277,398, 9., 22., and 23.4.26, and 10.1.27).—In the preparation of leuco-esters from vat dyes by the action of a metal, a tertiary base, and a substance (oleum, chlorosulphonic acid, sulphur trioxide, alkyl chlorosulphonates) which forms a sulphur trioxide compound of the tertiary base, it is found advantageous to add certain "assistants," namely, phthalic or succinic anhydride or imide, phosgene, ethyl chloroformate, acetyl chloride, benzoyl chloride, toluene-*p*-sulphonyl chloride, or other organic acid chlorides. The process is illustrated by 17 examples.
C. HOLLINS.

Manufacture of [sulphide vat] dyes. SOC. CHEM. IND. IN BASLE (GES. F. CHEM. IND. IN BASEL) (E.P. 270,348, 2.5.27. Switz., 30.4.26. Addn. to E.P. 199,360; B., 1924, 9).—In the thionation of indophenols from carbazole or *N*-alkylcarbazoles in presence of arylamines or their derivatives (cf. prior patent), markedly greener shades are obtained by adding urea to the sulphide melt.
C. HOLLINS.

Manufacture of stable preparations of vat dyes. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 259,999, 18.10.26. Ger., 17.10.25).—A dry vat dye, with or without a dispersing or wetting-out agent, is mixed with a dry alkali-starch preparation; a dry hyposulphite may also be added. The preparations remain unaltered when kept, and give clear vats on addition of water (or of hyposulphite solution if hyposulphite is absent), which contain no excess of alkali and are thus suitable for wool dyeing.
C. HOLLINS.

Production or development of azo dyes. H. T. BUCHERER (E.P. 249,526, 16.3.26).—Nitrosoamines (*anti*-diazotates) can be made to couple with coupling components by using a slightly alkaline medium at 90—100°. Strongly alkaline nitrosoamine printing pastes are nearly neutralised (*e.g.*, with calcium chloride) before steaming, the neutralising agent being added to the printing paste and/or to the fabric before printing.
C. HOLLINS.

Manufacture of an [azo] dye [for acetate silk]. SOC. CHEM. IND. IN BASLE (GES. F. CHEM. IND. IN BASEL) (E.P. 256,205, 14.7.26. Switz., 1.8.25).—Diazotised aniline, coupled with 1-*o*-chlorophenyl-3-methyl-5-pyrazolone, gives a yellow dye for acetate silk. The dye paste is preferably ground with a protective colloid, such as sulphite-cellulose liquor.
C. HOLLINS.

Manufacture of new intermediate compounds and of azo dyes therefrom. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, P. CHORLEY, and R. BRIGHTMAN (E.P. 277,756, 25.6.26).—Unsymmetrical carbamides are obtained by phosgenating equimolecular mixtures of 2-amino-8-naphthol-6-sulphonic acid (γ-acid) and an aminoacetanilide or a sulphonated amine of the benzene or naphthalene series (excluding sulphonated diamines, aminoformanilides, and aminonaphtholsulphonic acids), in presence of sodium acetate etc. The products when coupled with diazo compounds give dyes for cotton and regenerated cellulose silks. The product from γ-acid and *p*-aminoacetanilide couples with diazotised dehydrothio-*p*-toluidine to give a bordeaux, with diazotised *p*-chloroaniline a red, on viscose silk; with diazotised *p*-aminoazobenzenesulphonic acid a claret, and with diazotised *m*-sulphobenzeneazo-α-naphthylamine a blue, on cotton. The preparation and couplings of 8-hydroxy-1′ : 2-dinaphthylcarbamide-4′ : 6-disulphonic acid, and 8-hydroxy-2 : 2′-dinaphthylcarbamide-6 : 6′-disulphonic acid are also described.
C. HOLLINS.

Manufacture of pyrazolone-azo dyes. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 261,770, 19.11.26. Ger., 19.11.25).—Azo dyes are made by coupling a diazo compound with 1-(4′-chloro-6′-sulpho-3′-methylphenyl)-pyrazolones containing in the 3-position a methyl, carboxyl, or carbalkoxy-group. The preparation of fast yellow wool dyes from diazotised aniline-*o*-sulphonic acid, aniline, *p*-chloroaniline-*o*-sulphonic acid, and *o*-toluidine-3 : 5-disulphonic acid, and from tetrazotised $\beta\beta$-di-*p*-aminophenylpropane is described. The dye, *o*-toluidine-3 : 5-disulphonic acid $\rightarrow$ the pyrazolone-3-carboxylic acid, gives a very greenish yellow lake on barium sulphate.
C. HOLLINS.

Manufacture of *cyclo*trimethylenearylpyrazolones [1-aryl-3 : 4-trimethylene-5-pyrazolones]. C. MANNICH (E.P. 260,577, 18.10.26. Ger., 27.10.25).—The arylhydrazones of *cyclo*pentanone-2-carboxylic esters are cyclised by sodium alkoxides, forming 1-aryl-3 : 4-trimethylene-5-pyrazolones, which are also obtainable directly from the components in presence of sodium alkoxides without isolation of the hydrazones. 1-*Phenyl*- (m.p. 183—184°), 1-p-*bromophenyl*- (m.p. 200°), and 1-p-*tolyl*- (m.p. 202°) derivatives are described.
C. HOLLINS.

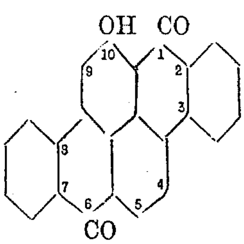
Manufacture of alkyl and aralkyl derivatives of *cyclo*trimethylenearylpyrazolones [1-aryl-2-alkyl- or aralkyl-3 : 4-trimethylene-5-pyrazolones]. C. MANNICH (E.P. 263,773, 1.12.26. Ger., 31.12.25).—By treatment with alkylating or aralkylating agents (*e.g.*,

methyl sulphate, ethyl bromide, β-bromoallyl bromide, or benzyl chloride, in presence of alkali) the products of E.P. 260,577 (preceding) are converted into compounds having antipyretic and analgesic properties. 1-*Phenyl*-3-*methyl*-3 : 4-*trimethylene*-5-*pyrazolone*, m.p. 128°, the 1-*phenyl*-2-*ethyl* compound, m.p. 143°, the 1-p-*tolyl*-2-*ethyl* compound, m.p. 118°, the 1-p-*bromophenyl*-2-β-*bromoallyl* compound, m.p. 109°, and the 1-*phenyl*-2-*benzyl* compound, m.p. 104°, are described. C. HOLLINS.

Manufacture of anthraquinone nitriles. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS & BRÜNING (E.P. 261,422, 15.11.26. Ger., 13.11.25).—Halogenated anthraquinones are heated with cuprous cyanide and an alkyl or aralkyl cyanide (particularly phenylacetonitrile). A complex compound separates on cooling or addition of alcohol, from which, by boiling with dilute nitric acid and removal of alkyl or aralkyl cyanide by steam-distillation, the cyano-anthraquinone is obtained free from copper. Halogen atoms in polyhalogenoanthraquinones may be replaced successively by cyano-groups. The preparation of 1 : 5-*dicyanoanthraquinone*, decomp. 380° ; 1-cyano-anthraquinone, m.p. 245—247° ; 8-*chloro*-1 : 4 : 5-*tricyanoanthraquinone* ; 1 : 4-*dicyanoanthraquinone*, m.p. 389—390° ; and 1 : 4 : 5 : 8-*tetracyanoanthraquinone*, is described. All the nitriles give green vats. C. HOLLINS.

Manufacture of *N*-dihydro-1 : 2 : 1′ : 2′-anthraquinoneazine. J. H. SACHS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,640,724, 30.8.27. Appl., 23.1.25).—An aqueous suspension of the leuco-compound is treated with air at 100° to give a product of increased purity. T. S. WHEELER.

Manufacture of benzanthrone derivatives. I. G. FARBENIND. A.-G. (E.P. 263,845, 29.12.26. Ger., 30.12.25. Addn. to E.P. 248,791 ; B., 1927, 550). — 2-Aroyloxybenzanthrones are converted by acid condensing agents (sodium aluminium chloride) into hydroxydibenzpyrenequinone derivatives. 2-Benzoyloxybenzanthrone, m.p. 215—216° (prepared by Schotten–Baumann reaction), gives at 180—240° 10-*hydroxy*-2 : 3 : 7 : 8-*dibenzpyrene*-1 : 6-*quinone* (annexed formula), the *methyl* and *ethyl esters* of which are yellow vat dyes. 2-p-*Toluyloxybenzanthrone*, m.p. 212—213°, similarly yields 10-*hydroxy*-4′-*methyl*-2 : 3 : 7 : 8-*dibenzpyrene*-1 : 6-*quinone* ; the *methyl ether* is a yellow vat dye. C. HOLLINS.



[Manufacture of] black sulphurised naphthol dyes. C. H. MARSCHALK (U.S.P. 1,641,182, 6.9.27. Appl., 11.3.24. Fr., 16.3.23).—See E.P. 212,936 ; B., 1925, 582.

[Production of aromatic sulphonic acids by] substituting halogen. K. SCHÖLLKOPF, Assr. to RHEINISCHE KAMPFER-FABR. G.M.B.H. (U.S.P. 1,645,974, 18.10.27. Appl., 16.8.23. Ger., 23.8.22).—See E.P. 202,975 ; B., 1924, 937.

V.—FIBRES ; TEXTILES ; CELLULOSE ; PAPER.

Analysis of cotton. Effect of disruption of the cotton hair on the extraction of fat, wax, and resin. R. G. FARGHER and L. HIGGINBOTHAM (J. Text. Inst., 1927, **18**, 283—287 T).—The effect of destroying the structure of the cotton hair is considerably smaller than was indicated by Knecht and Streat (B., 1923, 345 A), the extracts being due, not to material which cannot otherwise be removed, but to the easier removal of substances which are only slowly extracted from normal hairs. There is no evidence for the existence of relatively large amounts of wax and resin which are protected from attack by their position within the hair. B. P. RIDGE.

Deterioration of cotton during damp storage. A. C. BURNS (Ministry Agric., Egypt, Tech. & Sci. Service, 1927, Bull. No. 71, 92 pp.).—A full account of work previously described (B., 1925, 666). Spraying the unginned cotton with bactericides or fungicides might be used to prevent deterioration, but presents difficulties. Toluene, formaldehyde, and naphtha are too inflammable, whilst carbon tetrachloride has too powerful a solvent action, and, unless applied under strictly controlled conditions, affects the spinning qualities of the cotton. Hydrocyanic acid gas is probably the most suitable (except for poison risks to workmen), and comparative tests show that the spinning qualities of the treated cotton are unimpaired. B. P. RIDGE.

Influence of humidity on the breaking load of cotton at 20°. J. C. MANN (J. Text. Inst., 1927, **18**, 253—264 T).—Tests show that the strength of single cotton hairs increases with increasing humidity, the increase being of the order 10—15% of the breaking load at 44% R.H., and that above 66% R.H. the effect of humidity is negligible. B. P. RIDGE.

Mothproofing of fabrics and furs. L. E. JACKSON and H. E. WASSELL (Ind. Eng. Chem., 1927, **19**, 1175—1180).—By submitting a large number of possible moth-repelling chemicals to an exhaustive series of tests, the cinchona alkaloids and their derivatives have been found to be particularly effective as mothproofing agents. Application to the fabric is made by spraying or by immersion in a solution of the alkaloid. These substances possess the added advantages that they are inodorous, non-toxic to human beings, and non-injurious to fabrics. They adhere to the material on which they are used without being apparent, and may be employed in a variety of solvents by making use of salts of different solubilities. For use with solvents ordinarily employed in " dry-cleaning " plants, the fatty acid compounds are very suitable as, unlike the majority of the cinchona derivatives, they are readily soluble in these solvents. Quinidine salts have so far proved the most economical for industrial use. F. R. ENNOS.

Analysis of cotton. Action of hot dilute sodium hydroxide solutions on modified cotton cellulose. D. A. CLIBBENS, A. GEAKE, and B. P. RIDGE (J. Text. Inst., 1927, **18**, 277—284 T).—Oxycelluloses suffer a considerable fall of viscosity in cuprammonium solution after boiling for 6 hrs. with 1% sodium hydroxide,

whilst hydrocelluloses and normal bleached cotton are but slightly affected. The magnitude of this fall varies with the method of preparation of the oxycellulose, but is very great for neutral hypochlorite oxycelluloses—the most frequently occurring type of technically oxidised cotton. An exactly similar behaviour is shown when the alkali boil is replaced by the treatment given during the determination of copper number by Braidy's method. This test for the nature of the chemical attack of cotton is, therefore, conveniently made by comparing the viscosity of a sample with that of the material remaining after the determination of its copper number. The copper numbers of modified celluloses are reduced to one fifth of their original value by treatment of the material with boiling 1% sodium hydroxide solution for 6 hrs. under atmospheric pressure, and to about one tenth of the value by similar treatment at 20 lb./in.^{2} excess pressure. The Methylene Blue absorption generally rises as a consequence of such treatment, the rise being connected in some way with the concurrent fall in copper number. The percentage loss of weight suffered by modified cottons after treatment for 4 hrs. with boiling 1% sodium hydroxide under atmospheric pressure is about six times the copper number when the latter does not exceed 2·5, irrespective of the manner of modification. For values exceeding 2·5, hydrocelluloses suffer a greater, and oxycelluloses a smaller, loss of weight than corresponds to this relation, the divergence increasing with increasing cellulose modification.

B. P. Ridge.

Photographic method of investigating the colour of light sources, and the reflecting power of coloured fabric and other surfaces. P. W. Cunliffe and F. D. Farrow (J. Text. Inst., 1927, **18**, 291—302 T).—The method depends upon the production of equal densities on a photographic plate by exposures to a standard source of radiation (a gas-filled electric lamp) and to the lamp it is desired to investigate. Exposures are made for the same length of time, and the light falls on to the plate through an Ilford wedge screen and a spectral screen consisting of seven strips of Wratten gelatin filters, each passing a small range of wave-lengths. Attached to the spectral screen is a black slotted mask which allows light to pass through widths of 5 mm. of each filter. An exposure to the standard lamp is first made for one set of 5 mm. strips, and by a lateral movement of the mask these portions are covered and adjacent ones then exposed to the light from the unknown source, with the wedge reversed. The position of the point of equal density of the adjacent strips on the developed plate is found, from which, with a knowledge of the wedge constant, the relative intensities of any colour in the two exposures can be calculated. Undimmed lamps are found to be redder in the order gas-filled, vacuum, and carbon, the relative efficiency of the carbon lamp being low, and a marked loss of luminosity occurring when a gas-filled lamp is screened to give light of daylight quality. For the examination of the reflection from surfaces, a zinc white surface is taken as standard, and exposures are then made to light reflected first from the standard and then from the surface under investigation, the angle of incidence of light from a standard lamp to the surface being 45° and of reflection 0°. Results of the examination of both printed papers and dyed cotton cloths show the colour expressed in the form of curves, fading in the case of some of the dyed fabrics being indicated by a general increase in reflecting power. Reproducible results are obtained with both Wratten and Ilford filter screens.

B. P. Ridge.

Relation between fluorescence and the reddening of sulphite cellulose. VIII. Chemistry of the sulphite-cellulose digestion process. E. Hägglund and T. Johnson (Z. angew. Chem., 1927, **40**, 1101—1104).—The reddening of sulphite cellulose is due to the presence of lignosulphonic acid, and if this acid is eliminated from the carbohydrate portion by hydrolysis, the remaining material is no longer turned red by oxidation. The fluorescence of sulphite cellulose and of sulphite waste-liquor also depends on the presence of lignosulphonic acid, and there is a close relationship between the two phenomena. Cellulose which shows the strongest fluorescence also shows, on oxidation, the deepest red coloration, whilst that which no longer becomes red shows no violet fluorescence. The same group of the lignin which is responsible for the reddening also produces the fluorescence. This group, or constitutional ingredient, is formed or liberated through the sulphonation of the lignin during the cooking process, and, by treatment with oxidising agents, by air oxidation, or the action of alkalis or dilute acids at raised temperatures, it is irreversibly changed so that it no longer produces the luminous fluorescence. The latter must be distinguished from the violet colour which results from the treatment of wood with acids or acid-reacting liquids, or which appears after the removal of the violet fluorescence of the sulphite cellulose or the lignosulphonic acid by oxidation. This violet colour is changed to a fluorescent greenish-yellow by the action of alkalis. Neither the violet colour nor the greenish-yellow fluorescence appears when the cellulose ceases to give the red colour. The results stated apply not only to pine wood, but also to Scotch fir and to the sulphite celluloses obtained from both.

B. P. Ridge.

Cellulose in the paper and artificial silk industries. G. Raimondo (Notiz. chim. ind., 1927, **2**, 247—250).—Fir-wood cellulose, when boiled with 1% aqueous sodium hydroxide solution, reaches its maximum solubility, with loss of 18% by wt., in 2 hrs.; at the ordinary temperature in 16—20% sodium hydroxide solution, it lost 15·3% (maximal) in ½ hr. Digestion for 1 hr. with 18% sodium hydroxide solution should dissolve all the hemicellulose, even from straw and hemp. The following are values of the percentage dissolved by cold 18% and by hot 1% sodium hydroxide solution, respectively: German bleached, 15·1, 17·2; Czechoslovakian crude, 14·9, 17·7; Swedish crude, 15·8, 19·6; Norwegian bleached, 17·5, 19·2; Swedish bleached, 14·9, 17·3; poplar wood, 22·6, 22·5; esparto, 25·3, 21·2; straw, 38·5, 35·7; hemp stalk, 28·1, 23·8. The percentage soluble in boiling dilute sodium hydroxide solution is greater for soda or sulphite cellulose than for Italian (chlorine) cellulose.

Chemical Abstracts.

Grinding of immature eucalypts for mechanical pulp, and possibilities of manufacturing newsprint in Australia. L. R. Benjamin (Commonwealth of

Australia Council for Sci. and Ind. Res., 1927, Bull. No. 31, 37 pp.; cf. B., 1923, 923 A).—Mill tests show that eucalyptus chemical pulp when beaten sufficiently to ensure successful running on an ordinary paper machine gives a paper which, though of satisfactory strength, lacks the opacity, softness, and ink absorbency characteristic of high-grade newsprint. In view of these facts, attempts have been made to improve the finish of the paper by the addition of eucalyptus mechanical pulp. Small-scale grinding tests indicate that under carefully-controlled conditions of grinding the more important light-coloured eucalypts are capable of being converted into mechanical pulp of satisfactory colour and freeness. Laboratory papermaking trials showed that a furnish of eucalyptus sulphite pulp and eucalyptus mechanical pulp in the ratio 70 : 30, possibly with addition of a small quantity of long-fibred sulphite pulp, gave a paper somewhat stronger than newsprint, and closely resembling it in finish. No difficulty should be experienced in running such a paper on an ordinary paper-making machine, and estimated costs of production indicate that it should now be possible, especially with the assistance already proffered by the Australian Government, to establish a newsprint industry in Australia.

D. J. NORMAN.

Testing bleaching quality of wood pulp. H. L. JOACHIM (Zellstoff u. Papier, 1927, 7, 361—362).—For the rapid testing of bleaching quality an application of the permanganate method is described whereby results may be obtained in 20 min. and converted into terms of bleaching liquor by reference to a graph. The apparatus is a butter machine consisting of a thick glass jar of 2 litres capacity fitted with a mechanical stirrer with a curved glass rod substituted for the wooden paddle. A quantity of moist centrifuged pulp corresponding to 10 g. of air-dry fibre is stirred up with 1 litre of water, and the temperature is regulated in a constant-temperature bath to 37°, 25 c.c. of dilute sulphuric acid (1 : 4) are added, and then, without stopping the stirrer, 25 c.c. of *N*-permanganate solution are run in through the lid. The action of the permanganate is timed at exactly 7 min. at 37° with continuous stirring, and then by means of a burette inserted through the lid of the jar the excess of permanganate is discharged by *N*-oxalic acid added in portions of 0·5 c.c. at a time, at 30 sec. intervals until the end-point is approached. The correlation between bleach liquor and permanganate values is established by independent standardisation for different types of pulp.

J. F. BRIGGS.

Application of vat colours. WILSON. **Use of indicator-dyed wool.** KING.—See VI.

PATENTS.

Formation of colloid products. S. A. NEIDICH (U.S.P. 1,643,080, 20.9.27. Appl., 3.3.26).—Artificial threads consisting of a solid core and an outer tubular sheath are obtained by extruding a colloid filament containing more than 8% of hydrated cellulose into a sulphuric acid bath the acidity of which is at least 50% greater than is normally employed. The duration of the acid treatment is such that a complete skin is formed on the exterior of the thread leaving a colloid core in contact therewith. Reversion and dehydration of this colloid core are then brought about so that the core separates from the sheath to form an independent filament, whilst the outer sheath becomes shrivelled and thus capable of reflecting light with a scintillating effect.

D. J. NORMAN.

Washing or emulsifying agents [for textile material]. C. A. AGTHE (E.P. 253,554, 14.6.26. Ger., 13.6.25).—A normal or acid salt of pyrophosphoric acid is used, preferably in conjunction with known washing and emulsifying agents, as a washing agent for woollen, silk, and other soft fabrics.

D. J. NORMAN.

Machines for washing, milling, scouring, or otherwise treating fabrics with liquids. I. MITCHELL (E.P. 276,154, 3.9.26).—Two, or a series of, pairs of nipping rollers, driven at successively slower speeds, are arranged with their faces almost in contact so that the fabric is accommodated in the substantially enclosed space formed between each pair of rollers and the next.

D. J. NORMAN.

Washing or cleaning of wool. E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (E.P. 274,100, 3.5.26. Fr., 4.5.25. Addn. to E.P. 240,477).—The process of the main patent, dealing with the use of concentrated suint liquor for cleaning or reconditioning wool, is modified to eliminate the step of purifying the suint for re-use after treating wool in bulk or in the form of fleeces. This is effected by washing the wool, after a preliminary de-suinting operation if necessary, with sodium carbonate or soap solution. The wool is then treated with concentrated suint liquor on the counter-current principle, and is finally rinsed with water.

D. J. NORMAN.

Treatment [finishing] of fabrics, paper, etc. R. H. CHATHAM, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,640,596, 30.8.27. Appl., 18.7.21).—The material is padded with a solution of a cellulose ester or ether, *e.g.*, cellulose acetate, in a mixture of phenol, formaldehyde, and ethyl alcohol, then immersed in water to precipitate the cellulose derivative, and dried.

T. S. WHEELER.

Ornamentation of fabrics, leather, etc. BRIT. BEAD PRINTERS, LTD., J. C. VREDENBERG, and F. A. H. HEYNERT (E.P. 277,091, 8.6. and 13.7.26).—Carbamide–aldehyde condensation products in the plastic intermediate condition are applied to fabrics etc. and are converted into the hard, final product after surface-decorating materials, *e.g.*, glass particles, metal dust, pearl dust, mica flakes, have been introduced. Alternatively, the decorative materials may be incorporated with the plastic condensation product before its application to the fabric.

S. S. WOOLF.

Manufacture of cellulose derivatives. H. DREYFUS (E.P. 277,721, 26.3.26).—Cellulose is intimately mixed with alkali and an agent for removing water (calcium oxide) in presence of a diluent (benzene), and treated with ethylene oxide or chlorohydrin (2·5—5 mols. for each $C_6H_{10}O_5$ taken) preferably at 0—15°. In place of the ethylene oxide or chlorohydrin there may be used halogen esters of glycols, glycerol, and other polyhydric alcohols, or of ethers of these, also the products of action of hydrogen chloride on formaldehyde and acetaldehyde. The amount of water present should

preferably be not more than half the weight of cellulose; the alkali should be at least equal to and preferably 3—19 times the weight of water. Cellulose derivatives containing one or more free hydroxyl groups may be used in place of cellulose. C. Hollins.

[Non-inflammable] cellulose acetate compositions. S. J. Carroll, Assr. to Eastman Kodak Co. (U.S.P. 1,641,411—3, 6.9.27. Appl., [A, B], 27.4.25; [C], 21.12.25).—Cellulose acetate is rendered non-inflammable by addition of (A) 1-bromonaphthalene (5—30%), (B) bromobenzene or a monobromotoluene (5—50%), or (C) *s*-tribromoaniline or a homologue (4—16%). T. S. Wheeler.

Treatment of cellulosic materials. H. E. Potts. From Arnold Print Works (E.P. 276,877, 21.1.27).—Cellulosic material, particularly cotton, is superficially modified, *e.g.*, linenised, by padding it with caustic soda solution of such strength (*d* 1·095 or less) that, under the conditions of time and temperature of the treatment, caustic soda is adsorbed without causing any substantial mercerising effect, and then subjecting it to the action of, *e.g.*, cuprammonium hydroxide solution of such strength that, during the limited time of treatment given, it would of itself have no appreciable action on the cellulose. No substantial quantity of caustic soda diffuses from the cellulose during the operation, and the stability of the cuprammonium solution is not, therefore, adversely affected. D. J. Norman.

Production of cellulose and paper from straw, esparto, reed, and similar raw materials. E. L. Rinman (E.P. 269,154, 23.3.27. Swed., 7.4.26).—Esparto, straw, etc. are digested with caustic soda either alone or in admixture with sodium sulphide at 100—140°. The resulting pulp is separated from the black liquor at a temperature not exceeding 50° and washed with water at about 30°, these operations being carried out on a rotary filter with addition, if necessary, of high-boiling petroleum oils to prevent the formation of scum. In this way the glutinous substances present in the raw material are retained with the cellulose, and are present in the paper made therefrom. The washed pulp gives on beating a close sheet, and is suitable for making grease-proof papers, kraft papers (optionally with addition of 10% of long-fibred pulp), newsprint (in admixture with about 75% of mechanical pulp), etc. When bleached, which operation must be conducted at a temperature below 30°, the pulp may replace bleached sulphite pulp in fine papers of all descriptions. If it is found necessary in the manufacture of heavy greaseproofs to heat the pulp suspension to cause it to run more freely on the machine the temperature should not exceed 40°. D. J. Norman.

Manufacture of paper. H. A. Smith, Assr. to Hamersley Manuf. Co. (U.S.P. 1,644,451, 4.10.27. Appl., 15.8.25).—Paper containing hydrated cellulose (produced by beating) is impregnated with a solution of starch at 50—100°, pressed, and dried. D. J. Norman.

Treatment [separation of fibres] of paper pulp. C. R. Robinson, Assr. to Robinson Fibre Corp. (U.S.P. 1,644,447, 4.10.27. Appl., 6.2.25).—A solution of sodium peroxide is added to the pulp under conditions permitting free reaction of the peroxide; when the mixture is alkaline to litmus it is agitated to complete the separation of the fibres at a temperature somewhat above normal, but insufficiently high to injure the fibres. W. G. Carey.

Rendering articles moulded from wood pulp waterproof. J. J. H. Sturmey (E.P. 276,395, 19.4.26).—Containers and the like produced by the Drake process (cf. E.P. 115,587) are rendered waterproof by sizing the pulp in the beater, *e.g.*, with resin and aluminium sulphate, and subjecting the formed article, before removal from the mould, to a high temperature, *e.g.*, up to 400°, in order to fuse the sizing agent. D. J. Norman.

Production of artificial fibres, silk, hair, films, etc. from viscose. E. C. R. Marks. From Köln-Rottweil A.-G. (E.P. 277,716, 16.3.26).—See F.P. 612,879; B., 1927, 387.

Degreasing textile material. G. B. Ellis. From H. Kohnstamm & Co., Inc. (E.P. 276,121, 22.7.26).—See U.S.P. 1,598,305; B., 1926, 945.

Stencil sheet. S. Horii (U.S.P. 1,645,141, 11.10.27. Appl., 18.5.26).—See E.P. 250,798; B., 1926, 534.

Manufacture of strawboard. M. E. Pennington and A. B. Davis (U.S.P. 1,645,997, 18.10.27. Appl., 3.3.27).—See U.S.P. 1,625,090; B., 1927, 406.

Dyeing of cellulose esters and ethers (E.P. 258,611).—See VI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Recent advances in the application of [anthraquinone] vat colours. J. Wilson (J. Soc. Dyers and Col., 1927, **43**, 319—321).—Vat dyes may be applied to silk without deteriorating the fibre by the same methods as are used for dyeing cotton, and the resulting shades have excellent fastness, being unaffected by a degumming treatment (immersion in boiling soap for 8—12 hrs.). Vat dyes may be satisfactorily applied to wool from an ammonia-soda-hyposulphite vat such as is used in dyeing indigo. Dyeing is effected at 30—60°, the cold-dyeing colours (for cotton) yielding superior results when dyed on wool at 60°. An ordinary quality of wool is not deleteriously affected by immersion in solutions containing 4·5, 4·0, 3·5, 3·0, 2·5, 2·0, and 1·5 g./litre of caustic soda at 30°, 35°, 40°, 45°, 50°, 55°, and 60°, respectively; these quantities may be increased by 20% for a low-quality harsh wool, but should be decreased by 20% for a good-quality soft wool. Vat dyes may be applied to paper by adding the colour paste to the beater, followed by alum to promote exhaustion. The brightness of the resulting shade is proportional to the degree of fineness of the dye particles. For the colouring of oils and varnishes it is necessary to use vat dyes suspended in a fine state of division in non-aqueous medium such as linseed oil, turpentine, butyl alcohol, and ethyl acetate, since vat dyes after drying have but little tinctorial value when subsequently added to oils. The shade of a varnish coloured by the presence of a vat dye can be rendered lighter only by the addition of a white pigment and not by dilution of the varnish. A. J. Hall.

Use of indicator-dyed wool to control textile

operations. A. T. KING (J. Soc. Dyers and Col., 1927, **43**, 321—324).—Wool dyed with sulphonephthalein indicators (*e.g.*, Thymol Blue, Bromophenol Blue, Cresol Red, etc.), and also lacmoid, satisfactorily indicates the degree of acid or alkaline treatment to which it is subjected, although the sensitivity of these indicators when dyed on wool is much less than when used in solution. *E.g.*, Thymol Blue in solution suffers a colour change with 0·25% sulphuric acid, whereas 4% sulphuric acid is required to effect the same change when the indicator is dyed on wool. The indicators are suitably dyed on wool from a bath acidified with 1% of acetic acid, 0·02% of Phenol Red (or other indicators in proportion to their mol. wt.) yielding sensitive shades; 0·8% of lacmoid is used. The indicators dyed on wool have reasonable fastness to light, but the fastness to alkalis and acids varies, and the indicator may be removed from the wool by prolonged immersion in an acid or alkaline liquor. Indicator-dyed wool has yielded especially useful results in detecting faults occurring during the drying, scouring, and carbonising of wool materials. A. J. HALL.

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Process for bleaching and like purposes. C. B. THORNE (U.S.P. 1,642,978, 20.9.27. Appl., 6.12.24).—The disintegrated material is mixed with bleaching solution and fed into a bleaching tank of such design that, despite constant discharge during operation, the mass undergoing bleaching is not agitated.

D. J. NORMAN.

Dyeing of wool and/or silk. L. B. HOLLIDAY & Co., LTD., and C. SHAW (E.P. 277,833, 26.11.26).—Red-brown shades on wool, fixed by after-chroming or boiling with a solution of copper or iron sulphate, are obtained by dyeing from an acid bath with 2 : 5-dichloro- or 2 : 5-dibromo-*p*-benzoquinone. C. HOLLINS.

Dyeing of cellulose esters and ethers. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 258,611, 17.9.26. Ger., 17.9.25).—Primary disazo dyes suitable for acetate silk are obtained by coupling a mono- or di-hydroxy-compound of the benzene or naphthalene series with 2 mols. of diazo compounds, provided that the final product contains at least one free or substituted amino-group, not more than one sulphonic group, and not more than one naphthalene nucleus. Examples are: sulphanilic acid → phenol ← *m*-phenylenediamine (yellow). Resorcinol gives orange to brown dyes. C. HOLLINS.

Production of new fabrics [by effects on textile materials containing cellulose acetate]. BRITISH CELANESE, LTD., G. H. ELLIS, and R. J. MANN (E.P. 277,414, 15.6.26).—The cellulose acetate textile is treated locally (*e.g.*, by printing) with an aliphatic carboxylic acid and then steamed. In some cases (succinic and citric acids) delustring is accelerated by the acid; in others (acetic, propionic, chloroacetic acids) it is retarded. Hydroxy-acids are particularly advantageous, and formic, acetic, propionic, butyric, chloroacetic, aminoacetic, glycollic, lactic, citric, and succinic acids are specially claimed. C. HOLLINS.

Means for increasing the wetting and cleaning power of aqueous liquids used for treating fibrous materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 277,391, 10.3.26).—Sulphonic acids of polynuclear aromatic hydrocarbons, carrying as nuclear substituent a hydroaromatic radical with at least five carbon atoms, are used as wetting-out agents. In particular the sulphonated products of condensation of naphthalene with *cyclo*hexanol or methyl*cyclo*hexanols in concentrated sulphuric acid are used. C. HOLLINS.

Production of fast dyeings. F. PETERHAUSER, Assr. to DURAND & HUGUENIN S.A. (U.S.P. 1,645,925, 18.10.27. Appl., 12.3.25. Ger., 20.3.24).—See E.P. 231,189; B., 1926, 785.

Dyeing acidylcelluloses. P. RABE and W. SCHEPSS, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,645,450—1, 11.10.27. Appl., [A], 3.3.24; [B], 10.12.24. Ger., [A] 28.5.23, [B] 29.5.23).—See E.P. 216,838; B., 1925, 627.

Production of combined shades of azo and vat dyes on vegetable fibre. I. G. FARBENIND. A.-G., Assees. of G. PETZOLD and H. RITTNER (U.S.P. 1,640,802—3, 30.8.27. Appl., [A], 15.12.24; [B], 24.6.25. Ger., [A] 29.1.24; [B] 1.2.24).—See [A] E.P. 228,510 and [B] E.P. 228,514; B., 1925, 844, 845.

Stable preparations of vat dyes (E.P. 259,999). **Development of azo dyes** (E.P. 249,526).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Composition of commercial chemical lime. J. S. ROGERS (Ind. Eng. Chem., 1927, **19**, 1157—1158).—Analyses of 38 samples of lime representative of those used in various industries are given, and the results tabulated. A maximum limit of 1% of silica is not unreasonable, and a ferric oxide content of less than 0·5% is easily secured, whilst alumina in 65% of the samples was less than 1%. The results show that a lime free from phosphorus is possible, and that manganese, though found in nearly all the samples, is generally present in minute amount, sufficient, however, to affect the colour of the lime, giving it a greyish tint. Sulphur (as SO_3) varied from 0·04—3·33%, and may have been introduced or increased by burning the limestone with coal containing sulphur. The quicklimes varied from soft friable material to hard dense lumps, and in a few of the latter silica particles $\frac{1}{8}$ in. in size were present. Some quicklimes hydrated immediately, whilst others required 1 hr. or more, and generally the slaked products were soft, fine powders with less variation in colour than the corresponding quicklimes. W. G. CAREY.

Determination of potassium in mixtures of salts, especially in Chile saltpetre, by the perchlorate method. G. LEIMBACH (Caliche, 1926, **8**, 254—261).—For technical analysis, a single evaporation with hydrochloric acid (concentrated) and perchloric acid is sufficient. For more exact work the extraneous salts after the first evaporation are eliminated with alcohol (96%), and the residue is reprecipitated from water with perchloric acid; sulphates must be removed, but iodates do not interfere. In presence of boric acid an excess of perchloric acid is maintained.

CHEMICAL ABSTRACTS.

Unusual impurity in sodium bromide. V. ZANOTTI (Boll. Chim. farm., 1927, **66**, 513—514).—Attention is drawn to the presence of a phosphite as impurity in sodium bromide intended for medical use. The presence of sulphate is also detected. M. CARLTON.

Molten ammonium chloride. W. MERTNER (Chem.-Ztg., 1927, **51**, 638).—When ammonium chloride is heated at about 400° in a sealed tube it melts to a clear mobile liquid, which, on cooling, solidifies to a hard glassy mass similar in appearance to commercial "sublimed sal-ammoniac." This suggests that in the commercial method of making this salt true sublimation does not occur, but that, owing to the pressure inside the sublimation vessel, the ammonium chloride melts and then distils unchanged. A. R. POWELL.

Mechanism of the preparation of "sublimed sal-ammoniac." Molten ammonium chloride. W. SIEGEL (Chem.-Ztg., 1927, **51**, 759).—Polemical against Mertner (cf. preceding abstract). The pressure and temperature inside a sublimation vessel are much too low to cause any part of the substance to melt; the greater output of sublimed salt obtained by working with pressed tablets of crude salt is due to the greater surface obtained and to the greater ease with which the vapours can escape from the mass. Apparently the first sublimate consists of loose fibrous crystals, which, under suitable conditions of temperature and pressure, become coarser in texture and denser. A. R. POWELL.

Determination of the neutralising value of acid calcium phosphate [in baking powder]. C. W. HERD (Cereal Chem., 1927, **4**, 347—369).—The amount of alkali required for the neutralisation of acid calcium phosphate depends on the conditions of the experiment, owing to the fact that there is an equilibrium in solution between the mono-, di-, and tri-salts. The mono-acid salt has no neutralising power in the cold, but on heating equilibrium is gradually reached, so that the hot solution has a distinctly acid reaction. Direct titration of acid calcium phosphate with sodium hydroxide both in hot and cold solutions is unsatisfactory, as the end-point is dependent on the rate of addition of the alkali. The back-titration method involving addition of excess of alkali and titration of this excess with acid gives results in agreement with those obtained electrometrically, provided that the mixture is boiled until equilibrium is reached (about 10 min.), and that Thymol Blue or not more than 0·1 c.c. of a 0·2% solution of phenolphthalein is used as indicator. This method gives a value which is characteristic of the particular phosphate powder, but is too high for the commercial practice of the preparation of acid phosphate–bicarbonate mixtures for self-raising flour, since the mixtures based on this titration would be alkaline. Bailey's method of determining the p_H value of the actual baking ingredients may be used for checking the correct mixing of a raising powder, and can be used to correlate the above titration value with the most satisfactory acid phosphate–sodium bicarbonate ratio for baking practice. F. R. ENNOS.

Influence of form and proportion of lime used and of method of mixing on the resulting Bordeaux mixture. E. B. HOLLAND, C. O. DUNBAR, and G. M. GILLIGAN (J. Agric. Res., 1927, **34**, 677—686).—The use of different forms of calcium hydroxide in preparing Bordeaux mixture has been studied. Judging by the ratio of the volume occupied by the precipitate to the total volume of the liquid after 1, 2, and 3 hrs., it is concluded that, with chemically-precipitated calcium hydroxide, the best results are obtained if the equivalent of 1·5 lb. of calcium oxide are used with 4 lb. of copper sulphate and 50 gals. of water. Corresponding figures are 2·0—2·25 lb. for quicklime (made into milk of lime), and 2·0 lb. for commercial hydrated lime. The best method of mixing, similarly judged, is by pouring dilute copper sulphate solution into concentrated milk of lime, or the two simultaneously into a third receptacle. C. T. GIMINGHAM.

Dehydration of hydrated salts. IV. M. A. RAKUZIN and D. A. BRODSKI (Z. angew. Chem., 1927, **40**, 836—840; cf. B., 1926, 1011; 1927, 187).—When borax (undamaged crystals) is heated in an oven at 98° it is rapidly converted into the pentahydrate. The decahydrates of sodium carbonate and sulphate are completely dehydrated when treated in the same way, whereas the heptahydrate of magnesium sulphate loses only 4·5 mols. of water in 3 hrs.; of zinc sulphate, 6 mols. in 8 hrs.; of nickel sulphate, 4·5 mols. in 5 hrs.; and of cobalt sulphate, 6 mols. in 3 hrs. The pentahydrate of copper sulphate loses 4 mols. of water in 5 hrs., and potash alum 15 mols. in 2 hrs. A stream of dry air at 98° likewise completely dehydrates sodium carbonate, and removes 6 mols. from cobalt sulphate; it also brings about further dehydration of alum, 19 mols. in all being removed. Boiling ethyl alcohol removes 6 mols. from borax and from zinc sulphate, 8½ mols. from sodium carbonate, completely dehydrates sodium sulphate, removes only 2 mols. from magnesium sulphate, 2½ mols. from nickel sulphate, 2 mols. from cobalt sulphate, practically none from copper sulphate, and only 10 mols. from alum. The dehydration of borax by boiling alcohol depends on the strength of the latter. The products of dehydration by heating in air are crusty masses, which would need grinding before marketing. The various processes of dehydration are compared with one another from the economic point of view. W. A. SILVESTER.

Danger of explosion in the manufacture of bleaching liquor from liquid chlorine. E. SCHÖNBERG (Papier-Fabr., 1927, **25**, 581).—An explosion in an asphalted cast-iron tower in which a stream of milk of lime was treated with a counter-current of chlorine is attributed to the decomposition of calcium hypochlorite formed through supercooling of the reaction mixture. Calcium hypochlorite is unstable and, on warming, decomposes with the formation of chlorate and chloride, a reaction which may be completely without danger. In the presence of catalysts (*e.g.*, phosphorus, arsenic, sulphur, iron oxide) or of light, however, the reaction $Ca(OCl)_2 \rightarrow CaCl_2 + O_2$ takes place with explosive violence. If supercooling of the reaction mixture is avoided by ensuring complete vaporisation of the liquid chlorine before it comes into contact with the milk of lime, no danger of explosion exists. B. P. RIDGE.

Simple method for determination of active chlorine [in bleach liquor]. J. HAUSNER (Zellstoff u.

Papier, 1927, **7**, 363—364).—Although the use of sodium indigo sulphate as a quantitative measure of active chlorine in bleach liquor is not to be recommended, it may be employed to advantage as an indicator when mixed with the thiosulphate to a standard solution. For works' use the titration is carried out in a graduated stoppered cylinder having a zero mark near the bottom to indicate a known volume of bleach liquor; a drop of acetic acid is added to this liquor, and the blue-tinted thiosulphate solution is introduced gradually until the colour is no longer discharged. J. F. BRIGGS.

Ammonia as a source of nitrogen oxides for chamber acid plants. D. H. KILLEFFER (Ind. Eng. Chem.,1927, **19**, 1153—1156).—A description of American types of converter for the oxidation of ammonia in this connexion. Anhydrous ammonia is there used as frequently as aqueous ammonia as raw material. It is stored in tanks of 1¼-in. hammered steel, and vaporised by steam or by waste heat in a double-pipe heater, and then passes through an expansion valve to an air mixer. The heat-exchanger piping conveying the ammonia–air mixture is of aluminium, as iron has been found to catalyse the oxidation of ammonia to nitrogen under these conditions. C. IRWIN.

Behaviour of carbon and sulphur at high temperatures; [solid] carbon sulphides. J. P. WIBAUT (Z. angew. Chem., 1927, **40**, 1136—1137).—By the interaction of sulphur with amorphous or activated carbon at high temperatures, or by heating tetraiodothiophen, a solid heat-resistant carbon sulphide is obtained. This contains (approx.) 5% S, and is not an adsorption product, but its stoicheiometric composition is not yet established. The preparation of this and other solid sulphides of carbon is described. S. S. WOOLF.

Determination of carbon disulphide in its emulsions. H. J. FISHER (Ind. Eng. Chem., 1927, **19**, 1201—1202).—2 g. of emulsion are added to a tared glass-stoppered 100 c.c. flask containing 40 c.c. of 8% alcoholic potassium hydroxide. The solution is made up to 100 c.c. with 95% alcohol, and 5 c.c. are added to a mixture of 50 c.c. of water, 50 c.c. of 3% hydrogen peroxide, and 10 c.c. of 10% potassium hydroxide. It is heated for 1 hr., acidified, and the sulphur weighed as sulphate. Results given are about 96% of the theoretical assuming the purity of the carbon disulphide used. C. IRWIN.

Determination of sulphur trioxide in the presence of sulphur dioxide, together with some analyses of commercial liquid sulphur dioxide. J. R. ECKMAN (Sci. Papers U.S. Bur. Stand. No. 554, 1927, **22**, 277—285).—A nearly saturated and slightly acid solution of barium sulphite is used for precipitating the trioxide; the solution must be made and kept and the operations carried out in an oxygen-free atmosphere. Apparatus for carrying out the determination is described. Eight samples of commercial liquid sulphur dioxide were examined by this method; in all cases the gas obtained from the liquid was found to be free from sulphur trioxide, and only in three cases was the trioxide found in the residues. S. I. LEVY.

Corrosive sublimate as wood preservative. MOLL.—See IX.

Steels for ammonia synthesis. VANICK.—See X.

Absorption of nitrogen oxides by phosphate rock. MORRIS.—See XVI.

Ammonia by fermentation. WOODRUFF.—See XVIII.

Size of silver bromide particles. LOVELAND and TRIVELLI.—See XXI.

PATENTS.

Manufacture of sulphuric acid. Y. LE MONIÈS DE SAGAZAN (F.P. 618,560, 12.11.25).—Gases from pyrites or sulphur burners pass through apparatus comprising three sections in which they are brought in contact successively with nitrosulphuric acid and dilute sulphuric acid, with water or dilute sulphuric acid, and with sulphuric acid, *d* 1·71, for the recovery of oxides of nitrogen. L. A. COLES.

Production of concentrated nitric acid. C. UEBEL (G.P. 442,037, 12.1.26).—In a cyclic process for producing the acid by treating ammonium nitrate with concentrated sulphuric acid, the residual ammonium bisulphate, which may contain some nitric acid and water, is re-converted into ammonium nitrate by neutralisation with aluminium hydroxide with the addition of potassium nitrate in such quantity that potassium alum mixed with two equivalents of potassium sulphate crystallises out on cooling the solution. The crystalline product is treated with ammonia to recover aluminium hydroxide, potassium ammonium sulphate also being formed. Alternatively, the ammonium bisulphate is neutralised with ammonia in the presence of an equivalent weight of aluminium nitrate, whereby ammonium nitrate, aluminium hydroxide, and ammonium alum are formed. A further recovery process is described using manganic hydroxide or hydrated manganese dioxide, aluminium nitrate, and potassium nitrate. L. A. COLES.

Production of volatile acids, acid anhydrides, and chlorine. H. MEHNER (G.P. 442,166, 11.4.22).—Salts and other compounds destined for thermal decomposition, with or without the addition of steam, are sprayed (fused or in solution) through the hearth of a reverberatory furnace, and the heating gases are introduced elsewhere. The steam may be formed by the combustion of hydrogen or water-gas. The hearth is constructed of cement clinker for the decomposition of sulphates, and of loose heaps of fluorspar plaster for the manufacture of hydrogen fluoride. L. A. COLES.

Utilisation of waste acid liquors in chemical processes. C. A. KLEIN and R. S. BROWN (E.P. 277,769, 8.7.26).—Sulphuric acid obtained, for example, in the manufacture of titanium pigments (cf. E.P. 243,081; B., 1926, 99) is decolorised with animal charcoal, sulphur dioxide, or a small amount of a barium salt to carry down the colour with the resulting precipitate, and is then treated with barium oxide or carbonate to precipitate the acid as *blanc fixe* without precipitation of iron. A small quantity of a weak acid, *e.g.*, hydrochloric, nitric, or acetic acid, may be added to attack the oxide or carbonate before it acts as the precipitating agent. W. G. CAREY.

Production of commercial lumpy ammonium carbonate. I. G. Farbenind. A.-G. (G.P. 442,176, 17.3.25).—Ammonium bicarbonate, alone or mixed with ammonium carbamate or with ammonium carbonate, or a mixture of ammonia, carbon dioxide, and water in the correct proportions, is sublimed or heated under pressure.
L. A. Coles.

Hydration of lime. H. Dittlinger, Assr. to Dittlinger Crow Process Co. (U.S.P. 1,640,602, 30.8.27. Appl., 5.11.23).—Calcium hydroxide of increased plasticity is obtained by slaking calcium oxide with a dilute solution of potassium permanganate, sodium chloride, or similar compound. T. S. Wheeler.

Treatment of crude nitrate of soda. I. G. Farbenind. A.-G. (E.P. 266,744, 25.2.27. Ger., 25.2.26).—Oxides of nitrogen are obtained from crude sodium nitrate too low in nitrate to be economically worked, by heating it to red heat preferably in a current of air or other gas. The process is accelerated if gas which facilitates the decomposition of the nitrate is used, or if a small quantity of a non-volatile acid solid, *e.g.*, acid silicate, is added to expel the more volatile acids.
W. G. Carey.

Simultaneous manufacture of iron phosphide and fused cement. W. Kyber (E.P. 267,518, 8.3.27. Ger., 10.3.26).—Sufficient iron ore or scrap iron is added to a reduction melt or slag containing phosphates to combine with all the phosphorus, with the further addition of alumina, calcium oxide, and/or silica, the amounts depending upon the quantity already present and the required composition of the fused cement. Aluminium phosphate may be used as the primary material, in which case alumina is not added. W. G. Carey.

Production of zinc carbonate. N. A. Laury (U.S.P. 1,640,708, 30.8.27. Appl., 23.12.25).—Roasted zinc ore is leached with hot ammonium sulphate solution, zinc carbonate being recovered by cooling and saturating the resulting clear liquor with carbon dioxide.
T. S. Wheeler.

[Production of sulphur compounds from ores containing] sulphur. M. F. Coolbaugh and J. B. Read (Can. P. 264,357, 30.3.25).—The ore is roasted in air and the gases obtained, which contain sulphur dioxide, sulphur trioxide, and excess oxygen, are brought in contact with a chloride. Alternatively, a chloride is mixed with the ore before roasting. L. A. Coles.

Production of synthetic ammonia. Omnium des Ind. Chim. (Proc. Tocco & Landi), Assees. of L. Tocco and M. Landi (E.P. 262,090, 16.11.26. Fr., 27.11.25).—See Addn. to F.P. 607,059; B., 1927, 814.

Production of ammonia. Omnium des Ind. Chim. (Proc. Tocco & Landi) (E.P. 272,842, 17.11.26. Fr., 15.6.26).—See F.P. 607,059; B., 1927, 218.

Nitrating mixture (U.S.P. 1,640,737).—See XX.

Shadow producing agent (U.S.P. 1,644,446).—See XXI.

VIII.—GLASS; CERAMICS.

Properties of soda-lead oxide-silica glasses. S. English, W. E. S. Turner, and F. Winks (J. Soc. Glass Tech., 1927, **11**, 300—303).—In a series of glasses of the molecular type $6SiO_2,xPbO,(2-x)Na_2O$ and ranging from 72·86% SiO_2, 3·75% PbO, 22·62% Na_2O to 59·34% SiO_2, 27·77% PbO, 12·31% Na_2O, the annealing temperature decreased continuously from 478° to 446°, and the linear thermal expansion coefficient (25° to 90°) from 1090×10^{-8} to 842×10^{-8}, whilst the density at 25° increased from 2·5045 to 2·9960. From the results the coefficient of cubical expansion for lead oxide in glass was determined as $3 \cdot 18 \times 10^{-7}$.
A. Cousen.

Brittleness of opal glass. G. Gehlhoff and M. Thomas (J. Soc. Glass Tech., 1927, **11**, 347—358).—The brittleness of fluoride opal bulbs, as measured by impact strength, was found to be a function of wall thickness. With decreasing temperatures of working-out the glass, a rapid decrease of strength occurred over the range 1040—1030°, indicating 1050° as the limiting working temperature for the glass investigated. Confirmation was obtained by thermal resistivity tests involving the rapid chilling of spheres worked-out at varying temperatures. The glass showed a distinct heat absorption when, on heating, a temperature of 1050° was reached. Samples kept for some time at successively increasing temperatures and then rapidly chilled became clear and transparent at above 1050°, but opaque below this point. With three opal glasses of varying composition the critical zone occurred at points of approximately equal viscosity. A. Cousen.

Electrical conductivity of glasses at high temperatures. F. F. S. Bryson (J. Soc. Glass Tech., 1927, **11**, 331—346).—Conductivities were determined over a temperature range from the softening point of the glass to 1150°, by means of a high-frequency alternating current. In a series of soda-silica glasses the conductivity increased as soda substituted silica, and the greater the soda content the greater the increase in conductivity for a given rise of temperature. With soda-magnesia-silica glasses the conductivity increased as magnesia replaced silica. Soda-alumina-silica glasses progressively decreased in conductivity as the alumina replaced soda. With an apparatus devised for obtaining continuous records of conductivity of glass as it flowed from a tank furnace, variations were obtained which were in agreement with the limited variations of viscosity occurring under the system of control used in the works.
A. Cousen.

Noxious properties of [vitreous] enamels containing antimony. F. Flury (Z. angew. Chem., 1927, **40**, 1134—1136).—The effect of introducing various antimony salts into the food of rats, dogs, etc. is studied with a view of determining the desirability or otherwise of using enamelled cooking utensils containing antimony oxide or other compounds of antimony. The results are inconclusive, but the use of such articles is considered dangerous. S. S. Woolf.

Analysis of refractory materials. I. H. J. van Royen (Stahl u. Eisen, 1927, **47**, 1696—1697).—Silica in quartzite and silica brick is determined by loss in weight after the usual hydrofluoric acid treatment; the residue is dissolved in fused pyrosulphate and the iron determined by titration with titanous chloride. In a separate portion, also treated with hydrofluoric acid, the titanium and aluminium are precipitated together as

phosphate and weighed; the precipitate is dissolved in fused pyrosulphate and the titanium determined colorimetrically, the amount so found multiplied by 3·15 being subtracted from the weight of mixed phosphates to obtain the weight of aluminium phosphate. Manganese is determined by the Volhard method, and lime and magnesia as usual. The analysis of clays and grog is carried out in a similar manner, except that the sample is decomposed by fusion with alkali carbonate and the mixed aluminium and titanium phosphates obtained are redissolved in hydrochloric acid and reprecipitated to remove adsorbed sodium salts. Chromium should be tested for and, if found, the corresponding weight of chromium phosphate is deducted from the weight of combined phosphates. To determine titanium gravimetrically in this precipitate it is fused with a large excess of sodium carbonate, the mass leached with water, the insoluble sodium titanate collected and dissolved in hydrochloric acid, and the titanium precipitated by Gooch's acetate method. A. R. Powell.

Patents.

Manufacture of a highly refractory material. M. L. Freed, Assr. to U.S.A. (U.S.P. 1,644,244, 4.10.27. Appl., 10.6.26).—A mixture containing anhydrous aluminium silicate and topaz is calcined to convert it into mullite; the product is moulded with a temporary binder, and fired at a temperature sufficient to convert it into a homogeneous mass. W. G. Carey.

Manufacture of magnesite refractories. U.S. Metals Refining Co., Assees. of A. Marks (E.P. 271,847, 6.5.27. U.S., 6.5.26).—See U.S.P. 1,616,192; B., 1927, 189.

Drawing glass tubes or rods. S. G. S. Dicker. From N. V. Philips' Gloeilampenfabr. (E.P. 277,807, 30.9.26).

IX.—BUILDING MATERIALS.

Plastic magnesia [magnesium oxychloride] cements. L. G. Stewart (Ind. Eng. Chem., 1927, 19, 1139—1143).—Magnesium oxide for use with magnesium chloride as magnesium oxychloride cements is prepared by the calcination of magnesite or of precipitated magnesium hydroxide or carbonate; the product should not be "dead burnt," but must remain plastic and be very finely ground. Combined and adsorbed moisture should be below 5% otherwise the strength and setting time of the cement are adversely affected; free lime and calcium silicate are detrimental. Commercial magnesium chloride is used, and typical analyses of this and of various samples of plastic magnesia are given. Cement made from these materials with the addition of ground aggregate, sand, and asbestos are tested for strength, setting time, volume change, and also for weather- or water-resistance. The cement is used for building purposes on account of its high early strength, elasticity, resilience, thermal insulation, and fireproof qualities. W. G. Carey.

Use of plaster of Paris as an impression material. W. H. Sodeau and C. S. Gibson (Brit. Dental J., 1927, reprint, 27 pp.).—Whilst dental plaster consists mainly of the hemihydrate, the material is a complex and variable mixture, and the authors' conclusions are limited to certain types actually examined. The expansion on setting is about 0·3%, but is increased up to 1% by stirring after the mixture has appreciably stiffened or by addition of previously hydrated plaster. Expansion can, however, be reduced to a neglible amount by the use of a potassium salt as an accelerator, an alkaline substance such as borax being employed if necessary to prevent too rapid setting. The chloride, nitrate, or sulphate of potassium may be employed, the last in conjunction with borax being very suitable; it is convenient to add an appropriate dyestuff, such as Alizarin S. Data are given of tensile strengths developed by various mixtures under different conditions. S. I. Levy.

Importance of corrosive sublimate as a wood-impregnating material. F. Moll (Z. angew. Chem., 1927, **40**, 1137—1140).—Mercuric chloride and aniline or sodium derivatives of dinitrophenols are superior to copper sulphate and zinc chloride as wood preservatives, both as regards penetration and fungicidal effectiveness The supposed advantages of mixing sodium fluoride or other salts with mercuric chloride are considered, previous work in this field being summarised. In the fixation of mercuric chloride, the active product is usually mercury oxychloride and not calomel. The pre-eminence of corrosive sublimate as a wood preservative is supported by long-period practical tests. S. S. Woolf.

Commercial lime. Rogers.—See VII.

Patents.

Production of coloured effects in building materials. G. N. White (E.P. 277,389, 8.3., 26.7., and 18.8.26).—Vat dyes (and other coloured compounds capable of being vatted) in the form of dry powder, aqueous suspension, or colloidal solution, preferably after dispersion by oxidation of the leuco-compound in presence of a dispersing agent, are incorporated with building materials (cement, plaster, asbestos, lime, etc.), either by mixing dry, or in gauging water, or by subsequent impregnation. C. Hollins.

Manufacture of porous siliceous objects [from clay etc.]. F. C. and F. E. Kern (E.P. 262,826, 14.12.26. U.S., 14.12.25).—Wood or other cellular or fibrous material is heated for 10 min. at approximately 325° until its resilience is destroyed and ingredients more volatile than ketones are volatilised. The product (125 pts. by wt.) is mixed with 100 pts. of clay, rendered plastic by the addition of water, formed into shapes, dried, and burnt. Reactive agents such as light porous or colloidal siliceous substances may be added as a flux or to make the mixture more plastic. W. G. Carey.

Manufacture of a fibrous compound [for building purposes]. E. M. Lundgren (E.P. 276,709, 1.5.26).—Granulated cork, peat, or other vegetable material is artificially petrified by successive treatment with (i) calcium chloride solution and sodium silicate solution, with or without addition of lime water, (ii) calcium chloride solution and aluminium sulphate solution, or (iii) an aqueous suspension of calcium bicarbonate and lime water. The resulting products may be used as insulating or building materials. D. J. Norman.

Paper bags for containing cement, lime, etc. W. Ernst and E. Luh (Mittelbadische Papier-Manuf. Ernst & Luh (E.P. 278,275—6, 2.5.27. Ger., 7.12.26).

Fused cement (E.P. 267,518).—See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Stainless iron and its application to chemical plant construction. W. M. Mitchell (Ind. Eng. Chem., 1927, **19**, 1171—1175).—The true stainless steels containing 11—13% Cr and 0·3—0·4% C which require heat treatment are mainly suitable for valve seats, the wearing parts of crushers, etc., being too hard for most purposes. Mild stainless steel (11—14·5% Cr with under 0·12% C) can be obtained in all commercial forms and may be used for tanks and autoclaves. It is very suitable for valve stems, pump rods, and shafts. True stainless iron (above 16% Cr and below 0·1% C) is better suited for general purposes of fabrication. Its strength is not so great as that of the mild steels, but it is more resistant to corrosion and free from hardening tendency. Up to 0·1% Si is advantageous especially for resistance to nitric acid. Nickel–iron–chromium steels are more costly, but have the advantage of being easily welded. Chromium steel containing 25—30% Cr is useful when resistance to oxidation at very high temperatures is required. The high-carbon type is only available as castings. The proportion of chromium necessary to confer effective resistance to nitric acid is about 14%. At above 16% Cr the action of halide acids is slight. Sulphuric acid attacks all the alloys unless sufficient ferric sulphate is present in solution to inhibit the action, but " mixed acid " does not attack high-chromium alloys. Difficulties arise in both welding and rivetting except with nickel–iron–chromium alloys. Channels, angles, and seamless-drawn tubing can be successfully made of stainless iron. Sound castings are only readily obtained with high carbon content.

C. Irwin.

Surface decarburisation of carbon steels. E. H. Schulz and W. Hülsbruch (Arch. Eisenhüttenwesen, 1927, **1**, 225—240; Stahl u. Eisen, 1927, 47, 1694—1695).—The decarburising action of various atmospheres on three steels containing 0·5, 0·9, and 1·3% C has been determined at 750—1050°. In all gases, except pure nitrogen and carburising gases, such as coal gas, methane, or carbon monoxide, severe decarburisation takes place at 900° or above. Pure nitrogen and methane–hydrogen mixtures are inert, but even traces of carbon dioxide or steam in these gases causes severe decarburisation to occur. A similar action ensues if very small quantities of steam or carbon dioxide are present in gases which, if pure, act as carburising agents. In hydrogen or in a technical " reducing atmosphere," decarburisation commences at 550—600° and in steam, carbon dioxide, or air at 800°; in pure oxygen or perfectly dry air no action takes place below 850°. The depth of decarburisation in a given time increases in all cases with rise of temperature and, above 850°, with increase in the carbon content. The ferrite crystallites formed on the surface by decarburisation above 750° are extraordinarily coarse and columnar in shape, the long edges being perpendicular to the surface of the metal. In cases where a coating of scale is formed, this acts as a protective layer below 850°; the heaviest layer of scale is formed in a salt bath and only slightly thinner layers in oxygen or steam. A. R. Powell.

Report on metallic materials for electrical heating. [Analysis of nickel-chromium and nickel-chromium-iron alloys.] D. Harvey (Amer. Soc. Testing Materials, 1927, No. 20. Advance copy. 22 pp.).—In the determination of nickel with dimethylglyoxime, the residue insoluble in aqua regia, after removal of silica as silicon tetrafluoride, is fused with sodium carbonate, and a solution of the mass in hydrochloric acid is added to the principal solution. A 3% solution of dimethylglyoxime in 3% aqueous ammonia is used as the precipitant, the precipitate being dissolved in sulphuric acid and the nickel determined electrolytically. Chromium is determined by the persulphate method (permanganate being reduced by heating with sodium chloride in sulphuric acid solution), and iron by the cupferron method. For the determination of manganese the sample is dissolved in aqua regia and the solution evaporated with concentrated nitric acid. The manganese dioxide is precipitated with potassium chlorate, and dissolved in acidified ferrous sulphate solution, the excess of which is titrated with permanganate. Carbon is determined by Cain's method. The " insoluble residue " is obtained by treating the alloy (5—10 g.) with concentrated hydrochloric acid (200—400 c.c.) and nitric acid (1—2 c.c.) and removing silica as silicon tetrafluoride. The determination of silicon is conventional.

Chemical Abstracts.

Causes of failure in heat-resisting alloys. R. Sutton (Trans. Amer. Soc. Steel Treat., 1927, **12**, 221—234).—The lives of five case-hardening boxes of the 60% Ni, 20% Cr, 15% Fe type of alloy varied from 75 to over 7500 hrs. Four of the boxes containing aluminium were free from heavy scale. The principal reasons for failure were found to be strains caused by non-uniform castings, the presence of slag and other impurities in the alloy, also occluded gases caused by incomplete deoxidation. The best box consisted of uniformly distributed austenite and cementite, was non-magnetic, and had the lowest ball-hardness. It was poured at a low temperature and cooled slowly.

T. H. Burnham.

Magnesium and its alloys. J. A. Gann and A. W. Winston (Ind. Eng. Chem., 1927, **19**, 1193—1201).—Commercial magnesium prepared by electrolysis of the dehydrated chloride contains above 99·9% Mg. It forms numerous compounds with other metals, *e.g.*, silver, aluminium, copper, nickel, lead, tin, and zinc, with, in most cases, solid solutions as well; such alloys are hard and brittle. Their properties are usually improved by the addition of a third metal, *e.g.*, aluminium to magnesium–copper alloy. Manganese is introduced as a corrosion inhibitor. Particulars of the composition of four such commercial alloys intended for castings and forgings are given with their physical characteristics. They have d 1·74—1·84, and weight for weight are in most respects far superior to iron and steel. Metallographic specimens are etched with 2% nitric acid solution. The presence of aluminium and cadmium breaks up the magnesium–copper eutectic and gives a fine-grained structure. The metal is melted with a flux in cast steel pots, the flux covering the metal and protecting it. A mixture of magnesium chloride with sodium or potassium chloride is used, the density

of which is equal to that of the metal, so that the latter when melted floats as a ball within it. Magnesium oxide, sand, and dirt sink to the bottom. Oxidation of the metal during pouring is prevented by burning sulphur around it. Heat-treatment of certain cast alloys and before forging is desirable. The alloys can be extruded, hot-rolled, or machined. The corrosion which formerly occurred with magnesium and its alloys was mainly due to spongy metal and impure spots. Manganese is an inhibitor. Paint, varnish, or lacquer may be applied after a preliminary treatment with a hot acid phosphate solution. C. IRWIN.

Scleron metal. ANON. (Mitt. Materialprüf., 1927, **5**, 106—107; cf. B., 1927, 168).—A light aluminium alloy called scleron metal consists of 0·35% Si, 3·14% Cu, 0·49% Fe, 0·72% Mn, 13·20% Zn, 0·077% Li, and remainder aluminium. The properties of forged bars 39 mm. in diameter, after age-hardening, are *d* 3·0099, elastic modulus 725,000 kg./cm.^{2}, breaking strength 4890 kg./cm.^{2}, elongation 11·1%, and reduction in area 14·5%. Its resistance to corrosion by salt water equals that of pure aluminium. It is noticably free from segregation and internal oxidation, and should be suitable for constructional purposes. C. J. SMITHELLS.

Aluminium alloy, alneon, for castings. M. VON SCHWARZ (Z. Metallk., 1927, **19**, 390—395).—The name alneon is applied to zinc-aluminium alloys containing 10—20% Zn and small proportions of nickel and copper. The alloys have *d* 2·8—3·2, a Brinell hardness of 100—150, an elastic limit of 6·9—7·4 kg./mm.^{2}, a tensile strength of 20—34 kg./mm.^{2}, an elongation of 0·1—4%, and a high resistance to repeated impacts. The alloys are particularly suitable for the production of castings, owing to their small contraction on solidification and to the absence of any tendency to segregation. Like all aluminium alloys of this type alneon exhibits age-hardening properties, the maximum effect being obtained in 2—3 weeks at the ordinary temperature with alloys containing 80—87% Al, and in 2—3 months in alloys containing 88—90% Al. A. R. POWELL.

Tarnish-resisting silver alloys. L. JORDAN, L. H. GRENELL, and H. K. HERSCHMAN (Tech. Papers U.S. Bur. Stand. No. 348, 1927, **21**, 459—496).—The effect on the rate of tarnishing of silver in sodium polysulphide solutions of additions of the following elements singly or in pairs has been determined; aluminium, cadmium, copper, magnesium, zinc, silicon, tin, bismuth, antimony, tellurium, manganese, nickel, molybdenum, titanium, chromium, germanium, beryllium, gold, and sodium. The most resistant alloys were those containing zinc or cadmium, but their tensile strength was low, although they could readily be rolled into sheet. Addition of antimony or tin or of both to either zinc-silver or cadmium-silver alloys increased appreciably their strength, and slightly improved their resistance to tarnishing. The most satisfactory sterling silver alloys from the point of view of resistance to tarnishing, workability, and strength contained 4·5% Zn, 2% Sb, and 1% Sn, and had a tensile strength, after rolling into sheets, of 37,000 lb./in.^{2} Addition of gold to any of the alloys did not improve the resistance to tarnishing, but the tensile strength was increased. Alloys containing 15—20% Zn or Cd and 20% Au were highly resistant to corrosion, and of equal strength and hardness to standard silver alloys containing only copper. A. R. POWELL.

Preparation of alloy single crystals. H. SENG (Mitt. Materialprüf., 1927, **5**, 105—106).—A modification of Bridgman's method is described in which the molten alloy is sucked up from a crucible into a tube, tapered at the lower end and surrounded by a second tube to shield it from draughts. The whole is enclosed in a furnace which controls the temperature. The tube containing the alloy is lowered through the bottom of the furnace at the rate of 30 cm. in 24 hrs. Freezing begins in the tapered portion and the metal solidifies as a single crystal. Single crystals of a series of bismuth-antimony alloys were prepared 30 cm. long and 5 mm. in diameter. Although theoretically the crystals should vary in composition along their length this could not be detected by analysis. C. J. SMITHELLS.

Annealing metal wires. P. SIEBE (Z. Metallk., 1927, **19**, 385—389).—The rate at which wires 1·5 mm. thick of electrolytic and fire-refined copper, copper containing 0·6% Sn, brass with 67% Cu, commercial aluminium (99·3% Al), and soft iron are annealed in a nitrate salt bath at 200—800° has been determined and the results are reproduced in graphical form. With a standard time of immersion in the bath of 100 sec. electrolytic copper wires are completely annealed at 300—350°, refined copper wires at 400°, copper conductor wires (99·4% Cu) at 500°, brass wires at 450°, aluminium wires at 250—300°, and iron wires at 700°. The minimum time of annealing to obtain the maximum ductility is usually somewhat greater than that required to obtain the minimum tensile strength, especially at the lower annealing temperatures. A. R. POWELL.

Determination of antimony in white metals etc. H. R. FITTER (J.S.C.I., 1927, **46**, 414 T).—The sample is dissolved in nitric acid and the mixed oxides of tin and antimony are collected, washed, and dissolved in a hot solution of oxalic acid. The antimony is precipitated with hydrogen sulphide, the washed precipitate is dissolved in a 10% caustic soda solution, and traces of copper and lead are filtered off. The filtrate is acidified with oxalic acid, the antimony again precipitated with hydrogen sulphide, and the precipitate oxidised with fuming nitric acid and weighed as Sb_2O_4.

Preparation and application of beryllium. K. ILLIG (Z. angew. Chem., 1927, **40**, 1160—1163).

ERRATUM. B., Sept. 2, 1927, 657, col. 1, in the abstract on "Pure nickel and technical nickel," for "Mond nickel usually contains" read "a sample of Mond pellet nickel produced by the carbonyl process contained."

PATENTS.

Alloy impact-tool steel. P. A. E. ARMSTRONG (U.S.P. 1,645,213, 11.10.27. Appl., 14.11.25).—A very hard and tough steel consists of 1—2·5% W, 0·75—1·75% Cr, 0·5—0·6% Si, 0·5—0·6% Mn, 0·35—0·60% C, 0·05—0·95% Ta, the remainder being principally iron. F. G. CROSSE.

Manufacture of magnesium and the alkaline-earth metals such as calcium by the electrolysis

of molten chlorides. A. C. Jessup (E.P. 256,241, 27.7.26. Fr., 29.7.25).—In the electrolytic manufacture of magnesium, the electrolyte consists of a mixture of chlorides, including magnesium chloride, more electropositive than the latter and of such composition, *e.g.*, $3KCl,MgCl_2$, that the density is less than that of the molten magnesium formed. The cathode, which may be of graphite or of a metal slightly miscible with the metal formed and of high m.p., *e.g.*, chromium, tungsten, silicon, etc., or their alloys, is of such form that the metal produced flows over the cathode to maintain a clean surface "wetted" by the molten metal. The composition of the bath should be kept substantially constant.

C. A. King.

Improvement of magnesium alloys. I. G. Farbenind. A.-G. (F.P. 618,425, 2.7.26. Ger., 3.7.25).—Magnesium alloys containing 7—20% Al or 3—20% Zn or with a combined content of 3—20% (Al + Zn) together with small quantities of silicon and other metals are subjected to a sudden cooling during or immediately after casting. A. R. Powell.

Treatment of lead-tin alloys derived from waste material from lead-smelting works. M. Speichert, Assee. of F. Vogel (E.P. 253,908, 14.6.26. Ger., 17.6.25).—Residues accruing from lead-smelting processes, and containing 10—40% of impurities (chiefly copper, antimony, arsenic), are formed into electrodes and used as anodes in an acid solution of, *e.g.*, lead silicofluoride, at a suitable temperature. Lead and tin are dissolved and other impurities remain insoluble. C. A. King.

Resistance alloy. T. S. Fuller, Assr. to Gen. Electric Co. (U.S.P. 1,645,099, 11.10.27. Appl., 25.2.27).—The alloy consists chiefly of aluminium, together with 4—10% Mn and 2—8% Zn.

F. G. Crosse.

Apparatus for classifying ores etc. A. France (E.P. 264,536, 17.1.27. Belg., 18.1.26).—In a classifying apparatus in which a mixture of solid material and water is carried over a perforated surface to a discharging surface, the perforated portion is inclined at a different angle, or may be of varying angle as compared with the discharging surface. C. A. King.

Apparatus for galvanic processes, e.g., chrome-plating. Siemens & Halske A.-G. (E.P. 276,610, 16.10.26. Ger., 28.8.26).—All parts of apparatus coming into contact with the electrolyte, except the electrodes, are made of aluminium or aluminium alloy.

J. S. G. Thomas.

Treatment of aluminium to render it weldable. A. Kirchhof (E.P. 278,164, 16.9.26).—See U.S.P. 1,604,698; B., 1927, 47.

Lead-cadmium alloys. W. Friedrich, Assr. to Gen. Electric Co. (U.S.P. 1,645,098, 11.10.27. Appl., 12.4.24. Ger., 14.6.23).—See G.P. 405,148; B., 1925, 213.

Electrodeposition of tin. Vulcan Detinning Co., Assees. of H. R. McIlhenney (E.P. 269,474, 11.9.26. U.S., 14.4.26).—See U.S.P. 1,597,231; B., 1926, 922.

Chromium plating. H. C. Pierce, Assr. to Metals Protection Corp. (U.S.P. 1,645,927, 18.10.27. Appl., 5.3.26).—See E.P. 267,080; B., 1927, 390.

Refractory blocks [with tongue and groove] particularly for use in [roofs of] metallurgical furnaces. L. S. Longenecker (E.P. 278,070, 28.6.26).

Hardness testing device (E.P. 277,517).—See I.

Prevention of corrosion (E.P. 277,417).—See XI.

XI.—ELECTROTECHNICS.

Electrical insulating materials. A. Günther-Schulze (Z. Elektrochem., 1927, **33**, 360—368).—The electrical properties of gaseous, liquid, and solid insulating materials are reviewed and their theoretical interpretation is discussed. According to the purposes for which they are required, technical insulators can be divided into four groups. Examples of each type are described. The considerable effect of impurities in diminishing insulating power is emphasised.

H. J. T. Ellingham.

Electrolytic corrosion. Bolzinger.—See II.

Colour of light sources. Cunliffe and Farrow.—See V.

Conductivity of glass. Bryson.—See VIII.

Materials for electrical heating. Harvey.—See X.

Patents.

Electric furnace. J. A. Seede, Assr. to Gen. Electric Co. (U.S.P. 1,645,074, 11.10.27. Appl., 5.6.26).—A closed-loop heating channel having refractory walls communicates at widely separated points with a similar open-loop reservoir for the charge and contains an electric winding for heating, by induction, a conducting charge in the channel. J. S. G. Thomas.

Electric furnace. V. J. Chapman, Assr. to Gen. Electric Co. (U.S.P. 1,645,091, 11.10.27. Appl., 26.3.25).—A carbonaceous electrode ends within the wall of a refractory crucible, and a metallic terminal member having substantially the same electrical conductivity as the electrode is secured in electrically conducting engagement with the electrode at a point within the wall. J. S. G. Thomas.

Electric furnace. C. Steenstrup, Assr. to Gen. Electric Co. (U.S.P. 1,645,119, 11.10.27. Appl., 1.12.22).—An electric furnace comprises a container which is closed except at the bottom, a device forming a closure and seat for the container, means for moving the container relative to this device to permit introduction and removal of the charge, and means for maintaining a supply of any desired gas in the container in its closed and open conditions. J. S. G. Thomas.

Electric furnace resistor. T. A. Reid, Assr. to Westinghouse Electric & Manuf. Co. (U.S.P. 1,645,293, 11.10.27. Appl., 25.6.21).—A notched refractory bar is supported by some of a number of alined, spaced refractory members placed in certain of the walls enclosing a furnace chamber, and a second notched refractory bar is arranged in opposite parallel-spaced relation to the first bar, whilst a metallic resistor is helically wound round the two bars and supported in the notches of one of the bars. J. S. G. Thomas.

[Heating elements for] electric furnaces. British Thomson-Houston Co., Ltd., Assees. of A. N. Otis and

C. L. IPSEN (E.P. 258,276, 10.9.26. U.S., 10.9.25).—In electric furnaces of the type comprising refractory walls forming the heating chamber and apertures arranged at a level between upper and lower heating elements, elements having a downwardly-extending limb on either side are looped over supports arranged above each aperture, and sinuous heating elements are laid horizontally below. J. S. G. THOMAS.

[Separator for] primary cell. Manufacture of primary cells. M. L. MARTUS and E. H. BECKER (U.S.P. 1,644,344 and 1,644,389, 4.10.27. Appl., [A] 29.12.26, [B] 3.1.27).—(A) A separator for use in primary cells having a copper oxide cathode, a zinc anode, and a caustic alkali electrolyte consists of cellulose treated with a solution of sodium zincate (*d* 1·38). (B) A paper sleeve impregnated with sodium zincate solution and coated with a solution of alkali silicate is sprinkled, while still wet, with zinc dust and placed in a copper oxide–zinc cell with its metallised surface in contact with the copper oxide. J. S. G. THOMAS.

Electric battery. A. N. HAZLEHURST (E.P. 277,798, 13.9.26).—An electric battery comprises a positive element of lead peroxide, a negative element having an active surface of copper, an electrolyte consisting of sulphuric acid or a soluble sulphate, and means, *e.g.*, a layer of heavy oil floating on the electrolyte, for preventing access of air to the copper at the free surface of the electrolyte. J. S. G. THOMAS.

Preparation of an electron-emitting cathode. H. MILLER, Assr. to HAZELTINE CORP. (U.S.P. 1,640,710, 30.8.27. Appl., 1.3.26).—An alkaline-earth oxide or hydroxide colloidally dispersed in methyl alcohol is treated with carbon dioxide to form a colloidal solution of the corresponding carbonate, which is coated electrolytically or otherwise on platinum wire. The coating is converted into the oxide by heating at 1300°. T. S. WHEELER.

Method of artificially producing [increasing the radioactivity of] radioactive materials. A. GASCHLER (U.S.P. 1,644,370, 4.10.27. Appl., 4.9.24. Ger., 20.4.23).—Material submitted to a high temperature and pressure is treated with electric current at high potential. J. S. G. THOMAS.

Production of insulating materials [from minerals containing magnesium]. W. ZIMMERMANN (E.P. 277,577, 28.4.27).—Mineral substances containing magnesium, *e.g.*, dolomite, hornblende, are fused and then torn to threads in a stream of steam or compressed air. J. S. G. THOMAS.

Photometers [for electric incandescence lamps]. GEN. ELECTRIC CO., LTD., N. R. CAMPBELL, and C. G. EDEN (E.P. 277,467, 27.7.26).—Electric lamps of a similar type, even if of different powers, running at equal efficiencies, emit light of the same colour. Photoelectrically active metals vary in their sensitivity to different coloured light, *e.g.*, rubidium is relatively more sensitive to red than blue, compared with sodium. A photometer for determining the voltage at which electric lamps give a standard efficiency is constructed to make use of the above phenomena. B. M. VENABLES.

Electric [induction] furnace. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of M. UNGER (E.P. 263,857, 30.12.26. U.S., 31.12.25).—See U.S.P. 1,626,437; B., 1927, 450.

High-frequency electric furnace. F. GERTH, Assr. to C. LORENZ A.-G. (U.S.P. 1,645,526, 18.10.27. Appl., 24.3.26. Ger., 30.3.25).—See E.P. 249,843; B., 1926, 985.

Prevention of corrosion [due to stray currents]. E. CUMBERLAND (E.P. 277,417, 15.6.26).

Apparatus for analysis (E.P. 253,090).—See I.

Carbon granules for telephone transmitters (E.P. 277,129).—See II.

Magnesium (E.P. 256,241). **Lead–tin alloys** (E.P. 253,908). **Resistance alloy** (U.S.P. 1,645,099). **Apparatus for galvanic processes** (E.P. 276,610).—See X.

XII.—FATS; OILS; WAXES.

Setting point or titre [as an indication of the purity of fats]? M. DITTMER (Chem. Umschau, 1927, **34**, 258—259).—Polemical. Whilst the setting point of a fat and the titre of the fatty acid prepared from it are both determined in the same way, the probable error in the former determination is larger owing to the greater tendency of the fat to form supercooled solutions, and the result varies considerably with different forms of apparatus. The titre is therefore the more suitable criterion, but in those cases where the fat is to be used directly the setting point should be determined, using Shukoff's apparatus. W. J. POWELL.

Saponification of butter fat for determining the Reichert–Meissl value. G. SPITZER and W. F. EPPLE (J. Dairy Sci., 1927, **10**, 193—194).—Potassium hydroxide is added directly to the previously heated fat, and saponification is completed in an electric oven in 30—40 min. CHEMICAL ABSTRACTS.

Detection of blubber. J. DAVIDSOHN (Chem. Umschau, 1927, **34**, 260; cf. Tsujimoto, B., 1926, 1020; 1927, 754).—Tsujimoto's test for the recognition of blubber, using iodine trichloride, gave negative results with all samples tested. W. J. POWELL.

Mechanism of the sulphuric acid splitting of vegetable oils and distillation of the resulting fatty acids. A. BAGH (Chem. Umschau, 1927, **34**, 271—273). —A critical investigation has been carried out on the behaviour of cotton seed oil, a cotton seed oil–bone grease mixture, and the fatty acids obtained from them by various means, when examined in the Mackey apparatus. The mixed liquid fatty acids obtained from the sulphuric acid splitting of an 80% cotton seed oil–20% bone grease mixture gave a temperature of 99·5° after 2 hrs., the corresponding acids obtained by a potassium hydroxide saponification and subsequent liberation with sulphuric acid gave 147° after $\frac{3}{4}$ hr., whilst pure cotton seed oil gave 109° at 1 hr. Quantities of the semi-liquid acids from the sulphuric acid splitting of the foregoing mixture were examined after (1) steam distillation during $1\frac{1}{2}$ hrs., (2) heating at 200—210° for 12 hrs. in the presence of carbon dioxide and then distilling, (3) heating at 270—280° as in (2), and (4) heating with superheated steam at 280—290° for 35 hrs. and then distilling. The temperatures attained in the Mackey apparatus after 1 hr. were 144°, 152°, 98°, and 97°, respectively. It is concluded that

when a fatty acid mixture containing linoleic acid is heated at 280° in an atmosphere of carbon dioxide or steam, isomerisation occurs giving a form much more resistant to oxidation. This is apparent in the Mackey tests not only in the moderate temperatures attained, but in the lengthened period necessary for the temperature rise. E. Holmes.

Mackey test [for oils]. H. von Nabell (Chem. Umschau, 1927, **34**, 273—275).—Considerable errors may be introduced into the Mackey test by the use of metallic wire-gauze cylinders, which, in contact with the oil under test, give rise, particularly at the higher temperatures, to metallic soaps which have a marked accelerating effect on the self-heating properties of the oils. In an example, a sample of olein tested in an iron-gauze cylinder attained a temperature of 100° in 1 hr. 5 min., and ignited in 1 hr. 50 min.; an identical specimen, when tested in a copper cylinder reached 97° in 2 hrs., whilst a test carried out in a buckram cylinder gave less than 90° in 2 hrs. Buckram is, therefore, recommended as a suitable material for the purpose, or, failing that, one of the noble metals, as copper or silver. E. Holmes.

Various bromine-iodine values of oils and fats. W. Vaubel (Z. angew. Chem., 1927, **40**, 1143—1145).—The primary, secondary, and tertiary bromine-iodine values (cf. B., 1927, 49) of a number of vegetable and animal oils, fats, and waxes are tabulated and the main points of interest arising therefrom are discussed. As evidence of the selective nature of the reaction, secondary and tertiary values are shown by paraffin and glycerol, but not by benzene. In general, oils show increased values on saponification. Ring-splitting and oxidation processes are in the main responsible for tertiary values and partially for the secondary values, actual " additive " bromine corresponding to the primary value, *e.g.*, olive oil, or the secondary value, *e.g.*, linseed oil, or lying between the two. In the case of linseed oil, the three values are analysed in terms of the components of the oil, as stated by Eibner and Schmidinger. S. S. Woolf.

Determination of iodine-bromine numbers with potassium bromate and arsenite solution. L. W. Winkler (Arch. Pharm., 1927, **265**, 554—560).—In this determination it is an advantage to use approximately $0\cdot25N$-arsenious acid solution in place of the less stable $0\cdot5N$-sodium arsenite recommended in the Deutsches Arzneibuch. The arsenious acid solution may be accurately titrated against the potassium bromate solution alone, or in the presence of iodine as an indicator. If specially purified carbon tetrachloride is used there is no loss of bromine, either from the vapour phase or by leaving the reaction mixture for 24 hrs., and it is unnecessary to moisten the stopper of the flask with phosphoric acid. With ordinary carbon tetrachloride loss of bromine occurs. There is no necessity to use indigo-carmine as indicator, and the time of the reaction can be conveniently shortened to $\frac{1}{2}$ hr. It is then necessary to correct the limiting numbers given in the Arzneibuch to agree with this time of reaction (cf. Schulek, A, 1921, ii, 603). Good results are recorded with 12 unsaturated oils, including castor oil. The latter is an excellent substance for checking new test solutions; with a time of reaction of $\frac{1}{2}$ hr. the iodine-bromine number must be $84\cdot5 \pm 0\cdot5$. During the determination daylight must be rigidly excluded, and the amount of fat taken must be sufficient to react with approximately half of the bromine liberated. S. Coffey.

Gossypol content and chemical composition of cottonseeds during certain periods of development. W. D. Gallup (J. Agric. Res., 1927, **34**, 987—992; cf. Schwartze and Alsberg, B., 1924, 22).—Analyses of cottonseeds at different stages of development are reported. The percentage of gossypol present increased rapidly from the time the boll was mature until it opened, and continued to increase slowly for some time afterwards. No correlation was found between the formation of oil and the formation of gossypol. C. T. Gimingham.

Determination of free alkali in soap. J. Davidsohn (Chem. Umschau, 1927, **34**, 260).—In the determination of free alkali in soft soaps and in bar soap containing much water, the titration of the alcoholic solution with $0\cdot1N$-alcoholic hydrochloric acid is often uncertain owing to the return of the red colour of the phenolphthalein after the end-point is reached. This may be avoided by adding anhydrous sodium sulphate (4—6 g.) to the alcoholic soap solution (3·5 g. in 50—70 c.c. of 95% alcohol, previously neutralised), decanting, and washing the sodium sulphate with absolute alcohol, and then carrying out the titration. W. J. Powell.

Rubber seed oil. Ditmar.—See XIV.

Patents.

Separation of substances of dissimilar volatilities [*e.g.*, refining fats]. E. Wecker (E.P. 277,085, 7.6.26. Addn. to E.P. 213,267; cf. E.P. 235,792; B., 1925, 680).—The refining process described in the earlier patents is carried out in suitable apparatus, details of reaction chambers and scrubbing, heat-exchanging, and atomising devices being given. S. S. Woolf.

Reduction of auto-oxidation of oils and fats. A. Rechberg G.m.b.H., G. Braun G.m.b.H., and H. Oestermann (E.P. 254,303, 21.6.26. Ger., 24.6.25).—The auto-oxidation of oils and fats used for greasing wool is reduced or prevented by the addition of catalyst poisons (" paralysts "), *e.g.*, sulphur, arsenic sulphide. [Statutory ref. to E.P. 256,654; B., 1926, 887.] S. S. Woolf.

Fixing agent for perfumes etc. (G.P. 441,630).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Physical factors influencing the properties of paint pigments. A. de Waele (J. Oil and Colour Chem. Assoc., 1927, **10**, 232—249).—The specific absorptions of polar and non-polar media by pigments of various types and dispersions are tabulated, and these and typical photomicrographs are discussed with reference to the three types of solid-liquid dispersions previously postulated by the author, *i.e.*, colloidal dispersion, equilibrium, and flocculation. Permanently dispersed systems behave virtually as true fluids up to certain limiting volume concentrations, whilst flocculated systems show the effects of interference between dispersed particles, the stress-strain relationship being curvilinear and the

system as a whole showing static rigidity at close packing. The fundamental characteristics of such systems are bound up with the resistance set up by transverse orientation of liquid molecules from particle to particle.

S. S. WOOLF.

Application of vat colours. WILSON.—See VI.

PATENTS.

Method of imparting a decorative finish to articles. H. E. B. and J. H. GREENE (E.P. 276,864, 8.11.26).—Articles are dipped into paint, the medium of which contains a highly volatile solvent, *e.g.*, cellulose lacquers, and withdrawn slowly at an optimum rate. After superfluous paint has drained off, the article is revolved and reversed periodically until the film is set, the formation of folds or wrinkles being thus avoided. "Marbled" effects may be obtained by applying another paint of similar nature but different colour by dipping or otherwise at an intermediate stage in the drying of the first coat, and continuing the reversing and revolving process. S. S. WOOLF.

Manufacture of pigments, impregnating compounds, etc. R. M. GERLACH (E.P. 277,083, 7.6.26).—Heavy-metal salts of weak acids, *e.g.*, acetates, formates, ferro- or ferri-cyanides, silicofluorides of lead, mercury, and copper, are combined with the acidic components of resins, oils, or fats, *e.g.*, abietic acid, naphthenic acid, higher fatty acids, in the cold or with the application of slight heat, producing a complex compound which dissolves in excess of the acid solvent. Increased disinfectant, preservative, and fungicidal properties are claimed. S. S. WOOLF.

Manufacture of iron oxide. H. C. STEWART, Assr. to WESTMORELAND CHEM. & COLOR CO. (U.S.P. 1,642,975, 20.9.27. Appl., 3.5.23).—Dry copperas is fed into a furnace with a blast of air and burnt therein to oxide of iron. D. J. NORMAN.

Waterproofing composition. A. ASHBY (E.P. 277,051, 5.2.27).—Waterproofing compositions consisting of mixtures of turpentine or turpentine substitute, linseed or other drying oil, and 6—12% of a wax (preferably a mineral wax) are claimed. Small amounts of goldsize and/or driers may be added. S. S. WOOLF.

Condensation-product [air-drying] varnish. A. L. BROWN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,640,562, 30.8.27. Appl., 29.3.24).—A mixture of cresol, trioxymethylene, and tung oil is heated first at 100—120° and then, with addition of a drier, *e.g.*, cobalt linoleate, at 200°. The product is thinned with turpentine. T. S. WHEELER.

Manufacture of condensation products of crotonaldehyde. I. G. FARBENIND. A.-G., Assees. of W. KROPP (U.S.P. 1,640,899, 30.8.27. Appl., 5.3.26. Ger., 14.5.25).—The process claimed in G.P. 372,855 (B., 1923, 1140 A) is modified as regards its application to crotonaldehyde by omission of a condensing agent. T. S. WHEELER.

Waste acid from pigment manufacture (E.P. 277,769).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Preserved latex. V and VI. Preserving latex with ammonia. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, Buitenzorg, 1927, [17 and 18], 289—324, 325—346; Arch. Rubbercultuur, 1927, **11**, [8]).—V. To maintain latex in good condition in open tanks for 24 hrs. on the plantation the addition of 0·07% of anhydrous ammonia is sufficient. The decrease in alkalinity observed earlier in stored latex preserved with a sufficient proportion of ammonia is found to be due to loss by evaporation and not to any chemical or bacterial action; if the proportion of ammonia, however, is insufficient, the bacterial decomposition which leads to coagulation also leads to the formation of acids which cause a rapid decrease in the alkalinity. Spontaneous coagulation in ammoniated latex appears to be due to a decomposition of different type from that in non-preserved latex, the clotting in the former case occurring at a lower degree of acidity. The deposit which forms in freshly-ammoniated latex consists mainly of water, ammonium magnesium phosphate, and rubber, with smaller proportions of other mineral matter, protein, dirt, etc.

VI. The alteration in the rate of vulcanisation of the rubber in preserved latex on storage is not accompanied by any corresponding change in the alkalinity or in the nitrogen content of the rubber; the viscosity and plasticity remain almost constant and show a decrease only in old ammoniated latex. Rubber from ammoniated latex shows normal behaviour with respect to a slight decrease in viscosity and plasticity on storage; the tensile strength and slope remain constant, whilst the rate of vulcanisation gradually decreases and approaches the value for the control from unpreserved latex. With increase in the age of the ammoniated latex up to six months, the rate of vulcanisation and viscosity of the rubber decrease whilst its plasticity is constant, and its condition remains good even after 1½ years.

D. F. TWISS.

Isolation of the natural oxidation inhibitors of crude *Hevea* rubber. H. A. BRUSON, L. B. SEBRELL, and W. W. VOGT (Ind. Eng. Chem., 1927, **19**, 1187—1191).—Two substances of marked anti-oxidant character have been separated from the unsaponifiable portion of the acetone extract from raw rubber. These are high-boiling viscous oils of composition $C_{27}H_{42}O_3$ and $C_{20}H_{30}O$; they are apparently sterols and are present to the extent of 0·08% and 0·16% of the rubber, respectively. There are also present *n*-octadecyl alcohol, a ketone $C_{15}H_{24}O$, and a hydrocarbon $C_{15}H_{24}$, but these are without protective action. The aqueous extract of the acetone-soluble material also contains an anti-oxidant, probably a mixture of phenolic and ketonic substances. D. F. TWISS.

Micrographic studies of vulcanised rubber, showing the evolution of the free sulphur. REGNAUD (Chim. et Ind., 1927, **18**, 93—96).—Micrographic examination of various samples of vulcanised rubber at progressive intervals after vulcanisation reveals generally a succession of changes, viz., flocculation, coalescence, and crystallisation, in the residual free sulphur; the crystals in the interior tend finally to disappear as crystallisation proceeds at the surface. The observations are believed to support a colloidal theory of vulcanisation. D. F. TWISS.

Mould on rubber. O. DE VRIES (Comm. Proefstat. Rubber, Buitenzorg, 1927, [16], 262—283; Arch. Rubbercultuur, 1927, **11**, [8]).—The prolonged and stimulated growth of mould on raw smoked sheet rubber, *e.g.*, for several years, causes an actual loss of rubber hydrocarbon. Even after two years when the loss amounts to 20%, however, the rubber after cleaning and washing shows normal properties and composition, but continuous mould growth for five years, accompanied by a loss of over 30%, causes depreciation in viscosity, slope, and tensile strength, and a decrease in the rate of vulcanisation. The nitrogen content of the various samples indicates that the nitrogenous constituents are not consumed by the mould.

D. F. TWISS.

Ageing properties of rubber coagulated with formic acid. R. RIEBL (Comm. Proefstat. Rubber, Buitenzorg, 1927, [20], 354—359; Arch. Rubbercultuur, 1927, **11**, [8]).—Storage experiments over 2 and, in one case, 3 years give a definite indication that with formic acid as coagulant, the rubber obtained is comparable in properties with that produced with acetic acid. D. F. TWISS.

Comparative experiments with gas black and lamp black [in rubber]. P. SCHIDROWITZ (India-rubber J., 1927, **74**, 569—572).—Comparison of three samples of American gas black and three of European lamp black in standard mixtures of two types containing rubber (100 pts. by wt.) and black (36 pts.) with sulphur (4 pts.), zinc oxide (10 pts.), and diphenylguanidine (1 pt.), and with sulphur (5 pts.) and litharge (30 pts.), respectively, show that after vulcanisation the tensile product (tensile strength × elongation at break), energy of resilience, and resistance to abrasion are greater for the gas black samples. The differences persist after artificial ageing. D. F. TWISS.

Rubber seed oil and its industrial application. R. DITMAR (Caoutchouc et Gutta-Percha, 1927, **24**, 13,725—13,728).—After a review of earlier work on the oil present in the seeds of *Hevea brasiliensis*, this oil is shown to be more fluid than colza oil. Details are given for its conversion into light and dark "rubber substitutes." These, as well as the oil itself, are effective as softeners for raw rubber during the milling and mixing operations. D. F. TWISS.

Determination of sulphur in manufactured rubber. L. GRAFFE (Caoutchouc et Gutta-Percha, 1927, **24**, 13,722—13,723).—The sample (0·25 g.) in small pieces is treated with 5 c.c. of nitric acid (*d* 1·52), the reaction being initiated by cautious warming on a sand bath and subsequent removal. After the first vigorous action the heating is continued for ½ hr. A small quantity of a mixture of potassium chlorate and sodium nitrate (1 : 1) totalling 5 g. is introduced, and heating is continued until the disappearance of the chlorine. The reaction mixture is then transferred to a porcelain crucible with the aid of a few c.c. of nitric acid and heated on a water-bath with the occasional addition of portions of chlorate–nitrate mixture until no further reaction results. When at least 2·5 g. of the oxidising mixture have been added, the product is evaporated to complete dryness and the crucible cautiously heated over a flame until the oxidation process is complete. After introducing the remainder of the chlorate–nitrate mixture, the crucible is heated strongly until the evolution of oxygen ceases. The contents are then dissolved in water, acidified with hydrochloric acid, filtered if necessary, and treated with barium chloride in the usual way. D. F. TWISS.

Constitution of caoutchouc. R. PUMMERER (Z. angew. Chem., 1927, **40**, 1168—1172).—A review.

Rubber filter-press frames etc. FRITZ and CLARK, JUN.—See I.

PATENTS.

Process for accelerating the vulcanisation of rubber. W. SCOTT, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,639,903, 23.8.27. Appl., 14.8.24).—The product obtained by the action of a primary arylamine, *e.g.*, aniline (1—3 mols.), on an aliphatic aldehyde, *e.g.*, acetaldehyde (1—3 mols.), is treated at below 30° with 2% of concentrated hydrochloric acid, then neutralised, and heated with formaldehyde or acetaldehyde.

T. S. WHEELER.

Manufacture of aldehyde reaction products [accelerators] of the aldehyde derivative of a Schiff's base. C. O. NORTH, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,639,957, 23.8.27. Appl., 25.3.25).—The product obtained by the action of a primary arylamine, *e.g.*, aniline (2 mols.), on an aliphatic aldehyde, *e.g.*, acetaldehyde (3 mols.), is heated with formaldehyde or acetaldehyde at 95° under increased pressure for 4 hrs., and then under reduced pressure.

T. S. WHEELER.

Preparation of plastics containing rubber. J. MCGAVACK, Assr. to REVERE RUBBER Co. (U.S.P. 1,640,363—4, 30.8.27. Appl., [A] 3.7.23; [B] 24.3.25).—(A) Pale crêpe rubber dissolved in carbon tetrachloride is heated with sulphuric acid and an aliphatic aldehyde, *e.g.*, formaldehyde, with or without addition of phenol, at 100° for 2·5 hrs. (B) Vulcanised rubber dispersed in benzene is treated with chlorine, and then heated with phenol and formaldehyde. Substitutes for hard rubber are thus obtained. T. S. WHEELER.

Machine for mixing rubber and like material. FARREL & MACHINE Co. (E.P. 271,075, 9.5.27. U.S., 17.5.26).

XV.—LEATHER; GLUE.

Hydrolysis of pelt in acid media. G. GRASSER and S. TAGUCHI (Le Cuir, 1927, 426—428).—Pieces of pelt (1—10 g.) were immersed in 100 c.c. of solutions of hydrochloric, nitric, sulphuric, phosphoric, formic, oxalic, lactic, mono- and tri-chloroacetic, tartaric, citric, acetic, boric, and butyric acids of various strengths, and boiled under reflux. *N*- and 0·1*N*-hydrochloric, *N*- and 0·1*N*-nitric, and *N*-sulphuric acids hydrolysed the pelt completely in 1, 7, 1·3, 17, and 3·7 hrs., respectively; 0·1*N*- and 0·01*N*-sulphuric, *N*-phosphoric, 2*N*-formic, *N*-oxalic, lactic, mono- and tri-chloroacetic, tartaric, and citric acids hydrolysed the pelt completely after prolonged boiling. The hydrolytic action of the acids depends on their p_H value, and they can be arranged in the following order:—hydrochloric, nitric, sulphuric, oxalic, phosphoric, tartaric, citric, lactic, boric, butyric, and acetic acids. The first five exercise

strong hydrolytic action, tartaric, citric, and lactic acids are much feebler, whilst the remaining acids have very little effect. D. WOODROFFE.

Measurement of the adhesive strength of glue. C. E. LANYON (Ind. Eng. Chem., 1927, **19**, 1191—1193). —Briquettes were made by mixing 400 g. of sand with 60 g. of a 20% solution of the glue, filling into moulds, and drying for 6 weeks in air at 25° and 60% relative humidity. The adhesive strength of the glue was measured by determining the breaking strength of the briquette by means of a Riehlé cement-testing machine. The mean of a number of tests made by two investigators on the same glue differed only by 2%. No relationship was found between viscosity, tensile strength, and briquette strength, but on varying the acidity a maximum briquette strength was obtained with a glue solution of p_H 7·5. Wide differences were observed in the adhesive strength of glues of the same grade from different manufacturers, and hence the viscosity or jelly strength alone is useless for testing the suitability of glues for joints in the abrasive industry. F. R. ENNOS.

PATENTS.

Method of degreasing skins. C. A. BROWN and G. J. ESSELEN, JUN., Assrs. to MANUFACTURING IMPROVEMENT CORP. (U.S.P. 1,640,478, 30.8.27. Appl., 25.7.24). —Sheep skins are impregnated with an aqueous emulsion of petroleum and an olive oil soap, then immersed in warm brine, and finally pressed. T. S. WHEELER.

Ornamentation of leather (E.P. 277,091).—See V.

XVI.—AGRICULTURE.

[Exchangeable bases in soil.] A. M. SMITH (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1927, **B**, 8—9).— Treatment of soil with calcium chloride largely increased the proportion of exchangeable calcium at the expense of the other bases. An equimolecular solution of sodium and potassium chlorides increased the amount of exchangeable potassium to a much greater extent than the exchangeable sodium. A. G. POLLARD.

Exchangeable hydrogen and soil reaction. C. J. SCHOLLENBERGER (Science, 1927, **65**, 552—553).—*N*-Ammonium acetate solution at p_H 7·0 (prepared from ammonia and acetic acid, the reaction being controlled with a quinhydrone electrode) is preferred to ammonium chloride for extracting the exchangeable bases of soils. A method of electrometric titration is described. A. A. ELDRIDGE.

Sorption of nitrates in soils. K. FLEROV (Kolloid-Z., 1927, **43**, 81—84).—From the work of previous investigators it appears that nitrates are not adsorbed by soils. The author shows that this conclusion is due to neglect of differentiation between "true" and "apparent" adsorption. Experiments were carried out on the adsorption of potassium nitrate by gels of silica, ferric oxide, and alumina. Positive adsorption was observed in all cases, and the adsorptive power was greater the fresher and more rich in water the gel. Although the formation of insoluble basic ferric and aluminium nitrates may play some part, this is considered to be of a secondary nature. The true nature of the adsorption is the partition of the nitrate between the dispersion medium and the solid phase. E. S. HEDGES.

Absorption of nitrogen oxides in an aqueous suspension of phosphate rock. V. N. MORRIS (Ind. Eng. Chem., 1927, **19**, 1143—1147).—It was found that a suspension of ground Florida pebble phosphate in water or nitric acid absorbed oxides of nitrogen more effectively than water or nitric acid alone. The unabsorbed oxides of nitrogen were reduced to nitrogen with Devarda's alloy and the total nitrogen in the exit gases was determined. 50 g. of rock to 100 c.c. of 40% nitric acid gave the best absorption; excess of phosphate was injurious. Trials with calcium nitrate and with monocalcium phosphate produced a similar effect, the absorption–concentration curves showing maxima in each case. The dissolution of phosphate is slow under these conditions, and a large excess of acid or long time is necessary to bring it to completion. The mixture of calcium nitrate and monocalcium phosphate eventually obtained is rather hygroscopic for use as a fertiliser. C. IRWIN.

Effect of applications of [calcium] cyanamide on the nitrate content of field soils. F. E. ALLISON (J. Agric. Res., 1927, **34**, 657—662).—Experiments are reported which show that calcium cyanamide greatly retards nitrification in soils, the amount of nitrates present in soils receiving cyanamide being below that in untreated soils, even after 61 days' incubation. The results account, at least partly, for the poor results obtained with cyanamide as a fertiliser for cotton. C. T. GIMINGHAM.

Sodium hydroxide *versus* sodium carbonate [in soils]. P. S. BURGESS (Science, 1927, **65**, 445—446).— Few black alkali soils contain sodium carbonate under field conditions, the alkalinity being due to sodium hydroxide derived from hydrolysis of sodium zeolite. A. A. ELDRIDGE.

Determination of soil reaction by means of the quinhydrone electrode. E. BIILMANN and S. TOVBORG-JENSEN (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1927, **B**, 236—274).—Comparison of p_H determinations by means of the hydrogen electrode and by the quinhydrone electrode showed agreement to within p_H 0·1 in the majority of cases, the greatest difference observed being 0·2. The differences in values obtained with duplicate soil samples were from 0·1 to 0·3. Potential differences with the quinhydrone electrode were attained instantaneously, and there was no evidence of the presence in soil extracts of substances affecting the components of quinhydrone. No appreciable difference resulted whether the electrode was immersed in the soil–water paste or in the clear liquid above. The effect of changes in the soil : water ratio on the p_H values obtained is most marked in the case of soils with p_H less than 6·5, greater dilution causing higher values to be recorded. In many cases p_H values of fresh and of air-dried samples are substantially the same. Boiled or aerated water should be used in preparing soil suspensions, as the presence of carbon dioxide causes irregularities in the method. Nitrates, in the concentrations usually found in soils, do not appreciably affect the quinhydrone electrode. The latter cannot be utilised for determining p_H values greater than 8·5. A. G. POLLARD.

Quinhydrone electrode applied to the determina-

tion of p_H values of soils. C. Brioux and J. Pien (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1927, B, 5—7).—Using 1 : 5 soil-water suspensions, p_H values determined with the hydrogen electrode increase with the period of preliminary agitation. With the quinhydrone electrode maximum values are reached in a few minutes, and are markedly higher than those obtained with the hydrogen electrode. The p_H values of centrifuged soil extracts obtained with the quinhydrone electrode increase with the period of preliminary shaking, reaching a maximum after 20—44 hrs. The figures thus obtained are practically identical with those for soil suspensions determined by the hydrogen electrode.

A. G. Pollard.

Rapid determination of soil moisture by alcohol. G. J. Bouyoucos (Science, 1927, **65**, 375; cf. *ibid.*, 1926, **64**, 651).—Manipulative details of the hydrometer method are given. A. A. Eldridge.

Relation of potash removed by crops to active, total, acid-soluble, and acid-insoluble potash of the soil. G. S. Fraps (Texas Agric. Exp. Sta. Bull., 1927, No. 355, 33 pp.).—Methods are given for the determination of active, total, and acid-soluble potash in soil, and the relation between the form of the potash and the amount removed by crops is determined.

Chemical Abstracts.

Action of calcium carbonate and phosphorite on the composition of soil solutions and aqueous extracts from soils. D. Druzhinin (Nauk. Agron. Zhur., 1925, **2**, 629—644).—The accumulation of nitrates in unfertilised soils is ordinarily not accompanied by a decrease in the ratio of calcium (equivalent) to the total acid (equivalents); in limed soils and soils treated with phosphorite the calcium equivalent was approximately equal to the sum of the anions. Adsorbed calcium is the most important neutralising agent of the nitric acid. Chemical Abstracts.

Combined fraction of soil solution. Negative adsorption of electrolytes by soil. A. V. Trofimov (Nauk. Agron. Zhur., 1925, **2**, 613—628).— Calcium nitrate and chloride, and certain alkali chlorides, when added to black soils, loam, or podsol soils, become irregularly distributed in the soil solutions, being least concentrated in the surface soil solution as a result of negative adsorption. The degree of non-uniformity could be expressed by the adsorption isotherm.

Chemical Abstracts.

Saline county soils. Marion County soils. R. S. Smith, E. A. Norton, E. E. de Turk, F. C. Bauer, and L. H. Smith. **Will County soils.** R. S. Smith, O. J. Ellis, E. E. de Turk, F. C. Bauer, and L. H. Smith (Ill. Agric. Exp. Sta., Soil Rep., 1926, No. 33, 53 pp.; No. 34, 66 pp.; No. 35, 61 pp.).

Bordeaux mixture. Holland and others.—See VII.

Patents.

Drying of root-crops, grain, etc. B. J. Owen (E.P. 277,434, 18.6.26).—The material to be dried is piled between two concentric cylindrical structures, the inner one forming an internal air-distributing chamber. Heated air is delivered either into this internal chamber or into an external chamber formed by means of a removable air-tight casing, capable of being fixed round the outer cylinder. The chambers and supply-pipes are fitted with closing slides or shutters, and are so arranged that the direction in which the air is passed through the mass may be reversed at intervals or at any particular stage of the operation. C. T. Gimingham.

[Plant] disinfectant. R. Lieske, W. Schulemann, and W. Bonrath, Assrs. to Winthrop Chemical Co. (U.S.P. 1,640,901, 30.8.27. Appl., 28.1.25).—An organic mercury compound, *e.g.*, mercuribenzoic acid, is mixed with an alkali phenoxide in presence of water to give solutions of value as plant disinfectants.

T. S. Wheeler.

XVII.—SUGARS; STARCHES; GUMS.

Discoloration of sugars of different qualities at high temperatures in absence and in presence of other substances. O. Spengler and F. Tödt (Z. Ver. deut. Zucker-Ind., 1927, 623—640).—Comparison of samples of white sugar in respect of their liability to discoloration when melted, *e.g.*, for confectionery, can be satisfactorily made by heating 5 g. lots in test-tubes in an oil bath. With samples ranging in ash content from 0·0026 to 0·03%, the authors obtained very pronounced differences by raising the temperature from 100° to 167° and cooling again to 100° in a total period of 110 min. A study of the effects of moistening with 0·2 c.c. of various solutions before heating indicates that neutral salts and betaine are not responsible for discoloration, solutions of these substances producing no more effect than distilled water. A sample slightly discoloured by one heating is darkened much more by a second heating, so that the effect, as suggested by Lunden, is due more to decomposition products of the sugar than to foreign matters. Moistening with sodium hydroxide or mineral acid may cause strong discoloration, and with acid the colour depends very strikingly on the relative amounts of sugar and water present. Sugar (5 g.) moistened with 0·2 c.c. of 0·1N-hydrochloric acid melts below 100° to a clear, greenish yellow, partially inverted mass which darkens on continued heating and eventually becomes thoroughly caramelised. An inferior white sugar, which by itself cannot be melted without discoloration, will, if thus moistened with acid, yield a much lighter melt, which is partly-inverted, however, and therefore hygroscopic. If 5 g. of sugar are heated in a water-bath with 0·2, 0·6, 2, and 5 c.c. of 0·1N-hydrochloric acid, the maximum colour is produced by 0·6 c.c. These curious results with acid are attributed to changes in the activity of the acid, resulting from the abstraction of water by the sucrose and by the inversion process. J. H. Lane.

Influence of hyposulphites on [beet] syrups. J. Zamaron (Bull. Assoc. Chim. Sucr., 1927, **44**, 426—428).—In laboratory tests on beet syrups of about 65° Brix, treated with hyposulphite and then heated to 95° before filtration, no improvement in colour was perceptible with less than 1 g. of hyposulphite per litre, and the purity of the syrups was practically unchanged. Sulphited factory syrups treated with 40 g. of hyposulphite per hectolitre at 70° and then heated to 95° before filtration underwent a slight amount of inversion although alkaline. J. H. Lane.

Fermentation of beet molasses. CLAASSEN.—See XVIII.

PATENTS

Purification of sugar juice. P. J. T. MORIZOT (F.P. 609,731, 21.1.26).—The juice, after treatment with lime, is treated with carbon dioxide and phosphoric acid or a soluble monobasic phosphate. L. A. COLES.

Manufacture of starch. F. B. DEHN. From PENICK & FORD, LTD., INC. (E.P. 277,400, 19.4.26).—In the production of starch from corn, the overflow water from the gluten settlers instead of being rejected is used again—the minor portion for steeping the grain, and the major portion, after being sterilised by means of the steam obtained by concentrating the steep waters, for effecting further separation of the insoluble ingredients of the grain. F. R. ENNOS.

Manufacture of starch. E. C. R. MARKS. From CORN PRODUCTS REFINING CO. (E.P. 277,572, 8.4.27).—Successive quantities of maize are treated continuously by steeping and disintegrating the grain in the presence of sulphur dioxide, removing the germ, bran, and fibre, tabling the starch, separating the water from the gluten, and twice washing and filtering the starch with water containing sulphur dioxide to remove the soluble material. The water separated from the gluten and that from the starch filters is returned to the process for re-use, the former being used partly for steeping and partly in the germ and coarse slop separation, and the latter, after heating, in the fine slop separation. F. R. ENNOS.

Treatment of potatoes in the production of starch. MASCHINENBAU-ANSTALT HUMBOLDT (G.P. 441,911, 20.2.25).—Potatoes are treated in disc mills open to the whole circumference of the discs. The cells are completely broken down, and the starch may be easily washed out. B. FULLMAN.

Manufacture of large-grained dextrose. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,640,717, 30.8.27. Appl., 23.10.24).—See U.S.P. 1,521,830; B., 1925, 183.

XVIII.—FERMENTATION INDUSTRIES.

Fermentation of [beet] molasses by the aeration process of yeast manufacture. H. CLAASSEN (Z. Ver. deut. Zucker-Ind., 1927, 607—622).—Preliminary experiments to ascertain the losses of alcohol by evaporation during the aeration of 0·2—0·7% aqueous solutions of alcohol at temperatures of 25—31° showed losses ranging from 1·32 g. to 3·68 g. of alcohol per cub. m. of air. The worts for the fermentation experiments were made by diluting beet molasses to 20° Balling, heating to 80—90°, treating with 1% of sulphuric acid and 4% of superphosphate, and keeping the mixture for several hours to clarify. Only 5—10% of the total sucrose was inverted by this treatment, but inversion was practically complete within 4 hrs. of the addition of the yeast. In a series of small-scale experiments with 6-litre portions of wort, fermented with 30 g. of yeast, and differently aerated with 20—80 litres of air per min., the yeast crop increased with the intensity of aeration up to 60 litres per min., but 80 litres per min. produced no further increase. The total amount of nitrogen assimilated was the same in all cases, and the nitrogen content of the yeast formed was, therefore, in inverse ratio to the amount of the crop. In baking and keeping qualities the more intensely aerated yeasts were inferior to the others. Addition of 5 g. of ammonium sulphate to the wort, combined with aeration at 60 litres per min., did not improve the amount or quality of the yeast crop, although it increased the nitrogen content. A large-scale fermentation was carried out with wort from 3000 kg. of molasses, 120 kg. of superphosphate, and an extract of 525 kg. of malt germs. The amount of pitching yeast was 360 kg. (77% of moisture) and the duration of fermentation 11 hrs. Aeration was started at 3000 cub. m./hr., increasing soon to 5000 cub. m., and falling again to 2000—3000 cub. m. for the last 2 hrs. The crop of commercial pressed yeast was 1875 kg., representing 1733 kg. of new yeast containing 28·71 kg. of nitrogen or 48·6% of the original total nitrogen of the wort. Of the total sugar of the molasses, equivalent to 1575 kg. of invert sugar, 52·2% was accounted for as carbon dioxide, 26·8% as alcohol (including 6·2% lost by evaporation), 14·2% as non-protein matters of the new yeast, and the balance, 6·8%, as other undetermined metabolic products. The fermented wort contained unfermentable reducing substances equivalent to 1·4% of the original sugar. J. H. LANE.

Bacterial infection of beers. C. G. MATTHEWS and G. C. MATTHEWS (J. Inst. Brew., 1927, **33**, 502—506).—A deficiency of carbon dioxide in beers and stouts at any time during fermentation and the use of malt of poor quality in their manufacture predisposes such beers and stouts to infection. A case is described where a bottled beer, already so predisposed to adverse change, was attacked by ropiness, the cause of which was traced to the combined effect of the presence of *Pediococcus* in the finings and in the colouring solution. C. RANKEN.

Adaptation of the bactericidal action of chloroform to the preparation of bacterins. H. BUNYEA (J. Agric. Res., 1927, **34**, 623—630).—Experiments are reported on the use of chloroform as a sterilising agent in the preparation of bacterins. A broth culture of *Staphylococcus aureus* is sterilised by the passage of air containing chloroform vapour until the liquid has absorbed about 4·5% of chloroform, provided that the process is so retarded as to use that amount of chloroform in 2¾ hrs. C. T. GIMINGHAM.

Synthetic methyl alcohol and ammonia from butyl fermentation gases. J. C. WOODRUFF (Ind. Eng. Chem., 1927, **19**, 1147—1150).—The waste gases from the butyl alcohol–acetone fermentation of maize, which are very constant in composition at 60% of carbon dioxide and 40% of hydrogen, are stripped of their solvent vapour content (55% of acetone and 22·5% each of butyl and ethyl alcohols) by passing through cooled activated coconut-shell charcoal. After removal of the carbon dioxide, the hydrogen is mixed with air to give a 3 : 1 hydrogen–nitrogen mixture after burning the oxygen to water vapour, and this mixture is then passed to the high-pressure converter. The yield of ammonia is equal to that obtained from the purest electrolytic hydrogen. The high-pressure ammonia plant has been adapted to the production of methyl alcohol, for which purpose it is necessary to remove only a portion of the

carbon dioxide content of the fermenter gas, the reaction employed being $CO_2+3H_2 = CH_3\cdot OH+H_2O$. Investigations are in progress for the development of a suitable process for supplying carbon monoxide in order to make use of the reaction $CO+2H_2 = CH_3\cdot OH$, whereby the methyl alcohol output for a given compressor capacity may be increased 33%. F. R. Ennos.

Production of lactic acid by fermentation of wood sugar remaining after alcoholic fermentation. E. A. Marten, E. C. Sherrard, W. H. Peterson, and E. B. Fred (Ind. Eng. Chem., 1927, **19**, 1162—1165).—In the production of ethyl alcohol by the fermentation of sugars that result from the acid hydrolysis of wood, only a portion of the total sugar is acted upon by yeast. The residual sugars, consisting largely of pentoses, form about 35% of the total sugar from soft woods and 65% of that from hard woods. By the addition of suitable bacteria together with an excess of calcium carbonate and a nitrogenous material, *e.g.*, yeast, malt sprouts, dried blood, etc., a part of the residue may be made to undergo a second fermentation process, which is complete at 27° in about 10 days, with the formation of a mixture of lactic and acetic acids. The proportions of these acids obtained depend on the kind of sugars present in the liquor, and this varies with the nature of the wood and the extent of the previous alcoholic fermentation. The ratio of lactic to acetic acid is higher for soft woods (average 7·1 : 1) than for hard woods (average 3 : 1). Small-scale experiments indicate a yield, in addition to the alcohol, of 12 lb. of acetic acid and 84 lb. of lactic acid per ton of soft wood, the figures for hard wood being 32 lb. and 97 lb. respectively. F. R. Ennos.

Patents.

Manufacture of yeast. Internat. Yeast Co., Ltd. From Fleischmann Co. (E.P. 277,476, 24.8.26).—A fermenter is charged with wort of a gravity of about 2° Balling containing yeast nutrients sufficient to produce about half the weight of the pitching yeast. The wort is aerated during the fermentation, which is continued at 31—33° until the yeast content increases to about one and a half times the original yeast concentration. At that stage approximately one third of the wort is withdrawn and the yeast separated. The operation is repeated after adding to the fermenter sufficient water and nutrients to replace that withdrawn. C. Ranken.

Simultaneous dehydration and purification of alcohol. Soc. Anon. des Distilleries des Deux-Sèvres (F.P. 617,042, 3.6.26. Belg., 4.6.25).—Acids and esters contained in the alcohol are neutralised and hydrolysed, respectively, in the dehydrating column, or salts or organic acids formed in the process are withdrawn at the base of an auxiliary column. L. A. Coles.

XIX.—FOODS.

Rôle of phosphates in bread making. R. A. Barackman and C. H. Bailey (Cereal Chem., 1927, **4**, 400—410).—A survey of the work done on the investigation of the effect of phosphates on the colloidal properties of dough, and on yeast activity and reproduction. 0·2—0·5% of acid calcium phosphate improves the quality of a dough as is shown by the general improvement in baking quality, but further addition tends to impair it. None of the methods so far proposed gives a satisfactory measure of this improving effect. The addition of phosphate results in an increased rate of gas production, not by an increase in the number of functioning yeast cells, but because of the greater activity of each cell in fermentation. F. R. Ennos.

Interpretation of [bread] baking tests. L. W. Haas (Cereal Chem., 1927, **4**, 389—394).—The method for the production of the test loaves approaches actual baking practice as nearly as possible. Loaves are made from dough fermented for different times in order to gauge the correct fermentation period. A record is kept of the behaviour of the dough during fermentation in order to make any necessary adjustment in the absorption. The important points to consider in judging the test loaves, which are compared with those made from a standard flour, are measured loaf volume, oven spring, shape, bloom, grain, texture, colour of crumb, flavour, and odour. F. E. Ennos.

Determination of fat content of flour and milling stocks. C. W. Herd (Cereal Chem., 1927, **4**, 370—376).—Less fat is extracted by ether from completely dried flour than from that which is moist or partly dried. The first hour's drying, which removes at least 80% of the moisture, does not affect the amount of fat extractable by ether, but after the removal of the last 1—2% of moisture the amount extracted diminishes with the time of heating. On heating the extracted fat at 98° it becomes darker and less mobile, its refractive index changes from 1·4830 to 1·4890, and it becomes practically insoluble in the ordinary organic solvents. The low extractability of fat from partly dried flour appears to be due not to loss by "steam distillation" or to the formation of an insoluble oxidation product, but rather to polymerisation of the unsaturated acids of the fat. The change in the fat is not responsible for the gain in weight of a flour on heating after it has been dried out, since this occurs both in the extracted and in the non-extracted flour, but it probably accounts for the darkening in colour. A suggested method for the determination of fat in flour consists in drying for 1 hr. at 100°, extracting in a Soxhlet apparatus for 8 hrs. with anhydrous ether, filtering, evaporating to dryness, and drying the fat for 2 hrs. at 100°. F. R. Ennos.

Relationship between various factors and the ash constituents of milk. R. O. Davies and A. L. Provan (Welsh J. Agric., 1927, **3**, 236—249).—A close relationship was observed between the concentration of calcium and the yield of milk; the chlorine content varied widely, and the amounts of phosphorus and potassium were related to the activity of the glands. Low-protein winter rations should be adjusted to the same phosphorus content as the balanced ration. At the commencement of grazing there was a temporary increase in the concentration of phosphorus and protein. Chemical Abstracts.

Buffers of milk and buffer value. J. H. Buchanan and E. E. Peterson (J. Dairy Sci., 1927, **10**, 224—231).—The calculated buffer value of milk varies with the

p_H range; its average value at p_H 8·5—9 is 0·0067. Casein has little effect as a buffer in the region of its isoelectric point, and the phosphates are least among the important buffers of milk. CHEMICAL ABSTRACTS.

Comparison of the Babcock, Gerber, and Roese-Gottlieb methods for determining fat in milk and cream. A. C. DAHLBERG, G. E. HOLM, and H. C. TROY (New York Agric. Exp. Sta. Tech. Bull. 122, 1926, 13—32).—The Babcock and Gerber methods are equally and sufficiently accurate; the Roese-Gottlieb method is variable. CHEMICAL ABSTRACTS.

Acid and fat content of [technical] caseins. J. MARCUSSON and M. PICARD (Mitt. Materialprüf., 1927, **5**, 122).—The standard methods of Lunge and Berl, Höpfner and Jaudas, and Ulex for determining the acid content of technical casein give discordant results, and the following is recommended. Casein (5 g.) is mixed into a paste with 5 c.c. of water, and, after 15 min., is ground with quartz sand, extracted with ether, and the extract titrated against phenolphthalein with alcoholic potash solution. A low value is obtained if the treatment with water is omitted. Calculating the acid content as lactic acid is inaccurate, as the free fatty acids, which have 3 times the mol. wt., vary in amount. If the ether solution is extracted 3—4 times with water the lactic acid can be determined by titrating the aqueous solution, and the fatty acids by titrating the ether solution, assuming a mean mol. wt. of 280. The ether solution is now extracted twice with water to remove the soaps, the ether distilled off, and the residue of neutral fats weighed. Analyses of 5 samples of casein gave lactic acid 1·6—3·9%, fatty acids 0·8—4%, and neutral fats 0·8—4·2%. C. J. SMITHELLS.

Effect of ethylene on the composition and colour of fruits. E. M. CHACE and C. G. CHURCH (Ind. Eng. Chem., 1927, **19**, 1135—1139; cf. B., 1924, 149, 439, 726).—During the colouring of citrus fruits by ethylene (1 pt. of the gas in 5000 pts. of air) there is no apparent change in the composition, as shown by the sucrose content and acidity of the edible portion. Ethylene in a concentration of 1 in 100,000 is a much more effective colouring agent for lemons than 1 pt. of propylene in 5000. In the case of dates no changes are observed which could not be brought about by heat alone. The action of ethylene on persimmons is to accelerate the colouring both inside and outside, to remove the astringency, and to soften the fruit. No satisfactory indications of colouring have been obtained by the ethylene treatment of bananas, tomatoes, pomegranates, and avocados. F. R. ENNOS.

Preservation of fruits in sulphurous acid solutions. W. V. CRUESS and A. H. EL NOUTY (Fruit Prod. J. Amer. Vinegar Ind., 1927, **6**, No. 11, 18—20).—Corrosion of the container takes place if canned maraschino cherries contain more than 25 mg. of sulphur dioxide per kg. For the preservation of fruits in sulphur dioxide solution, more than 1500 mg. per litre must be present initially. CHEMICAL ABSTRACTS.

Variations in the chemical composition of cabbage and sauerkraut. W. H. PETERSON, E. B. FRED, and J. A. VILJOEN (Canner, 1925, **61**, No. 4, 19—21).—Variations according to the variety and maturity of cabbage are: sugar, 3—4·2; total nitrogen, 0·15—0·24; soluble nitrogen, 0·06—0·16; calcium, 0·034—0·057%. With increasing maturity the sugar decreases and the nitrogen increases. Values for sauerkraut are: moisture, 89·6—91·5; sugar, 0·09—0·77; titratable acid as lactic, 1·44—1·93; volatile acid as acetic, 0·28—0·42; non-volatile acid as lactic, 1·13—1·52; ethyl alcohol, 0·29—0·61%. The amino-nitrogen content of the sauerkraut is much higher than that of the cabbage. CHEMICAL ABSTRACTS.

Determination of total sulphur in [animal] tissues and in foods. L. LEMATTE, G. BOINOT, and E. KEHANE (Ann. Chim. analyt., 1927, [ii], **9**, 296—297).—Balland's method of evaporation with sodium carbonate, calcination, and extraction of the residue with acidulated water, the sulphur being finally precipitated as sulphate and weighed, gave low values when applied to lentil starch. Accurate results were, however, obtained by a method in which the material was digested with a mixture of perchloric acid and concentrated nitric acid. The oxidation of the organic sulphur is very difficult, and the use of a less concentrated nitric acid may give very low results. G. A. ELLIOTT.

Crude fibre in food. E. J. MAGERS (J. Amer. Dietet. Assoc., 1925, **1**, 73—77).—Determinations were made of crude fibre and moisture in 23 foods in various conditions. Different samples of the same food showed wide variations. CHEMICAL ABSTRACTS.

Phosphates in baking powders. HERD.—See VII.

PATENTS.

Manufacture of nutritional beverages and food products. F. H. PECK (E.P. 254,724, 30.6.26. U.S., 1.7.25).—A beverage rich in active vitamins is made by a modification of the process of making beer, in which materials containing soluble vitamins, *e.g.*, lucerne, clover bran, vegetables, etc., are added towards the end of the boiling stage, the brew being exposed to ultra-violet rays subsequent to the stages of sterilising and of fermentation. F. R. ENNOS.

Pectin preparations and manufacture of preserves and jellies. DOUGLAS PECTIN CORP., Assees. of H. G. LOESCH (E.P. 262,736, 3.11.26. U.S., 10.12.25).—2—10% of powdered pectin is suspended in a saturated sugar solution containing a suitable amount of organic acid, *e.g.*, tartaric or lactic acid, with the addition of flavouring and colouring materials if desired, whereby the pectin is wetted but not dissolved. For use, the above mixture is diluted with hot water or fruit juice to dissolve the pectin, and more sugar is added so that the liquid contains sufficient pectin, acid, and sugar to ensure jelly formation. F. R. ENNOS.

Vessel for mixing chocolate etc. H. W. HICKLEY, Assr. to BAKER PERKINS CO., INC. (U.S.P. 1,645,990, 18.10.27. Appl., 19.11.23. U.K., 21.11.22).—See E.P. 203,587; B., 1923, 1054 A.

Mixing machines for chocolate or other material. A. SONSTHAGEN (E.P. 278,152, 2.9.26).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Contact poisons, especially in the catalytic formaldehyde process. A. SIEGL (Chem.-Ztg., 1927, **51**, 782—783).—Loss of activity of the metallic catalyst used in the oxidation of methyl alcohol to formaldehyde is frequently due to traces of some contact poison in the air, and is detected most readily by control analyses of the issuing gases, being shown by a steady increase in the total percentage of the gaseous carbon compounds (carbon monoxide and dioxide, and methane). Halogen compounds are particularly effective poisons, producing marked loss of activity even when present in the air in such small quantities that their presence cannot readily be detected analytically. If possible, the use of purer air drawn from the open at a distance from the works is recommended, and will soon repay the cost of the necessary air conduit. A. B. MANNING.

Occurrence of α-propylene glycol. K. SCHUTT (Oesterr. Chem.-Ztg., 1927, **30**, 170—171).—When crude sugar is fermented in the presence of sodium sulphite, glycerol is produced together with a little trimethylene glycol, the latter passing over first when the mixture is vacuum-distilled by means of superheated steam (Connstein and Lüdecke, A., 1919, i, 463). A sample of these first runnings has been converted into the diacetates and carefully fractionated. Approximately 14% consists of α-propylene glycol diacetate, m.p. 185—187·5°, which gives rise to the α-dibromide, m.p. 141—143·6°. E. HOLMES.

Determination of mercury salicylate and lactate by a mercurimetric method. A. JONESCO-MATIU and C. BORDEIANU (Bul. Soc. Chim. România, 1927, **9**, 11—16).—The salicylate is oxidised by heating with a mixture of 1 pt. of nitric acid and 2 pts. of concentrated sulphuric acid until the solution is clear. After cooling and diluting, 10% potassium permanganate solution is added until a faint colour persists, followed by a small excess of sodium nitroprussiate. The solution is then titrated with standardised 0·1*N*-sodium chloride until the muddy colour disappears. Determinations on the pure salts give results ranging from 99·2% to 100·45%. When oxidising the lactate heating is unnecessary, and results of the order 99·49—99·91% are obtained. F. S. HAWKINS.

Commercial applications of chlorophyll derivatives. F. M. SCHERTZ (Ind. Eng. Chem., 1927, **19**, 1152—1153).—Zinc and copper phæophytins, obtained by substitution of these metals for hydrogen in the derivative resulting from the treatment of chlorophyll with acid, are more stable than the natural chlorophyll. The copper compound is unaffected by acids or alkalis and retains its bright green colour on exposure to light or after being stored in various oils for as long as two years. It is used in foods, soaps, etc. both to hide and to give colour. A water-soluble product, a soapy mass containing a copper salt of chlorophyllin, and alcohol- and oil-soluble products, consisting of fat and wax mixed with copper phæophytin, are used for colouring purposes. Chlorophyll and its derivatives in the form of phæophytins exhibit marked therapeutic properties and are used in increasing amount in pharmaceutical preparations. F. R. ENNOS.

Determination of atropine in pills. O. EHRISMANN (Arch. Pharm., 1927, **265**, 547—549).—Quantities of atropine too small to be determined chemically were determined by the mydriasis produced in cats. Four animals were selected and their sensitiveness to atropine was determined; this varied from 0·00018 to 0·00027 mg. Pills containing known small amounts of atropine were extracted with either warm water or ether, which removed the alkaloid. The latter was separated, dissolved in water, and the aqueous solution diluted until a drop (27 mg.) no longer produced mydriasis. The amount of atropine extracted was then determined from the volume of solution. The results obtained were never below 60%. The ethereal and aqueous extracts gave identical results. S. COFFEY.

Isolation of nicotine from *Nicotiana attenuata*, Torr. J. F. COUCH (Amer. J. Pharm., 1927, **99**, 519—523).—The isolation and chemical and pharmacological identification of nicotine from *N. attenuata* is described. Determination of the total alkaloid, calculated as nicotine, by Young's method (B., 1927, 123) gave the following amounts in the dry samples: leaves 1·45%, stems 0·48%, roots 0·25%. Non-volatile alkaloids were absent. The lethal dose of nicotine injected intraperitoneally in guinea pigs is 17—20 mg./kg. E. H. SHARPLES.

Chemical test of nicotine dusts. R. M. HIXON and C. J. DRAKE (Iowa State Coll. J. Sci., 1927, **1**, 373—377).—Thatcher and Streeter's method (New York Agric. Exp. Sta. Bull., 1923, No. 501, 3) is untrustworthy, since it measures the rate of evolution of nicotine vapour plus the rate of decomposition of the nicotine. With bentonite as a carrier, 23—86% of the nicotine was unaccountably lost in 24—48 hrs.; a residual oil may be oxynicotine. The concentration of nicotine vapour produced is approximately proportional to the nicotine content of the dust except with magnesium carbonate as a basis, when evaporation is hindered. Nicotine is more toxic than hydrogen cyanide. CHEMICAL ABSTRACTS.

Ergot oil. H. MATTHES and P. SCHÜTZ (Arch. Pharm., 1927, **265**, 541—546).—The oil from *Secale cornutum* of German origin had d^{20} 0·9210, n_D^{20} 1·4694, n_D^{40} 1·4623, iodine value 70·09, saponif. value 195·4, $[\alpha]_D^{15}$ +10·5°, unsaponifiable matter 1·01%. The mixed fatty acids, consisting of 28·79% of solid and 70·24% of liquid acids, had m.p. 40·5—41°, n_D^{40} 1·4550, iodine value 70·46, $[\alpha]_D^{15}$ +2·74°, mol. wt. 281·7, acetyl value 69·82 (acetyl value of ethyl esters 73). The solid fatty acids consisted of palmitic acid and higher homologues. The liquid acids, consisting of 50% of hydroxyoleic, 45% of oleic, and 5% of linoleic acids, on bromination and treatment with light petroleum, b.p. 30—60°, furnished an insoluble, liquid *dibromohydroxyoleic acid*, which afforded *hydroxyoleic acid*, n_D^{20} 1·4721, n_D^{40} 1·4684, mol. wt. 298·6, iodine value 85·6, acetyl value 142·9, on reduction with platinised zinc. The petroleum solution of bromo-acids contained only linoleic acid tetrabromide, m.p. 114°, and dibromo-oleic acid. Another sample of oil of Austrian origin gave, d^{20} 0·9170, n_D^{20} 1·4710, n_D^{40} 1·4642, α_D^{15} +10·6°, iodine value 66·6, unsaponifiable matter 1·037%. The fatty acid mixture contained 27·1% of solid and 71·2% of liquid acids, the latter furnishing the hydroxyoleic acid with an acetyl value of 144·6. S. COFFEY.

Genus *Mentha*. XIII. Oil of *Mentha piperita*, L., produced in 1924. S. M. GORDON (Amer. J. Pharm., 1927, 99, 524—530).—An oil from *Mentha piperita*, L., had d^{25} 0·9088, n 1·465, α —13·25°, acid value nil, ester value 29·22, ester value after acetylation 154·7, esters 10·32%, total menthol 49·52%, combined menthol 6·46%, free menthol 43·10%. Only 13% of the free menthol could be separated from the oil by freezing. The isolation of *d*-menthone, *l*-menthone, *d*-pulegone, *l*-α-pinene, and a terpinene is described. Phellandrene and limonene were absent.

E. H. SHARPLES.

Properties and constituents of an oil extracted from the seeds of *Digitalis purpurea*. I. S. MELLANOFF (Amer. J. Pharm., 1927, 99, 549).—The amber-coloured, semi-drying oil from the seeds of *D. purpurea* has n^{20} 1·4755, $d^{15\cdot5}$ 0·9231, acid value 9·30, saponif. value 207·5, ester value 198·2, unsaponifiable matter 6·12%, soluble fatty acids 1·66%, insoluble fatty acids 90·0%, hydroxy-acids nil, liquid acids 75·8% (n^{20} 1·4670), solid acids 9·2% (n^{20} 1·4685), and iodine value (Hanus) 127·9. E. H. SHARPLES.

Methyl alcohol by fermentation. WOODRUFF. **Lactic and acetic acids by fermentation.** MARTEN and others.—See XVIII.

PATENTS.

Concentration of dilute aqueous formaldehyde solutions. M. MUELLER, Assee. of A. ZIMMERLI (Can. P. 264,342, 10.9.24).—The dilute solution is heated under reflux until equilibrium is reached between formaldehyde, its polymerides, and hydrates, when the mixture is fractionated. B. FULLMAN.

Manufacture of alcohols. R. B. MACMULLIN and R. E. GEGENHEIMER, Assrs. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,641,544, 6.9.27. Appl., 27.8.24).—An alkyl, *e.g.*, methyl, chloride is passed with steam (1 mol.) over calcium hydroxide at 400° and a pressure of 13 atm., the time of contact being about 40 sec.

T. S. WHEELER.

Manufacture of alkyl sulphates. R. H. MCKEE (U.S.P. 1,641,005, 30.8.27. Appl., 21.5.23. Renewed 13.7.27).—An aliphatic alcohol, *e.g.*, ethyl alcohol, is treated with sulphuryl chloride (1 mol.) at below 30°, excess of chloride being removed by distillation.

T. S. WHEELER.

Nitration process and nitrating mixture. G. B. TAYLOR and A. S. RICHARDSON, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,640,737, 30.8.27. Appl., 15.7.22).—The solution of nitric and nitrosylsulphuric acids in sulphuric acid, obtained by treatment of sulphuric acid with nitrogen peroxide, can be employed to nitrate organic compounds, *e.g.*, benzene. The spent acid, consisting chiefly of nitrosylsulphuric and sulphuric acids, is treated with steam or sulphur dioxide to form nitric oxide, which is re-oxidised to the peroxide.

T. S. WHEELER.

Production of tertiary nitriles. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 253,950, 22.6.26. Ger., 22.6.25).—Nitriles of the type $CHR_2{\cdot}CN$ are alkylated etc. by means of sodium and an alkylating agent. α-Ethyl-*n*-butyronitrile, $CHEt_2{\cdot}CN$, is treated with sodium or potassium in dry ether, benzene, xylene, etc. and the product is warmed with allyl bromide to form *αα-diethyl-Δ^γ-n-pentenonitrile*, b.p. 78°/9 mm., which may be hydrolysed to the *amide*, m.p. 74°. Benzyl bromide similarly gives *α-benzyl-α-ethyl-n-butyronitrile*, b.p. 120—122°/1 mm. (*amide*, m.p. 75°). C. HOLLINS.

Increasing the yield of civetone, starting from civet. M. NAEF and F. FIRMENICH (M. NAEF & CIE.) (E.P. 267,893, 20.1.27. Switz., 17.3.26).—The civetol which accompanies civetone in civet may be oxidised to civetone by usual oxidation methods. *E.g.*, civet is hydrolysed with alkali and the neutral oil, with or without previously removing civetone as semicarbazone, is oxidised with chromic acid, or catalytically over a metal catalyst at 250—300° *in vacuo*. C. HOLLINS.

Manufacture of aqueous emulsions of paraffin for use as medicines. E. GLÜCKSMANN (E.P. 255,456, 13.7.26. Ger., 14.7.25).—Stable, stiff emulsions of paraffin in water are formed in the presence of vegetable gums and algæ as joint emulsifying agents. *E.g.*, a 70% emulsion is formed by the addition of 70 kg. of paraffin to a solution containing 1 kg. of agar-agar, 25 kg. of water, 0·5—1 kg. of gum arabic swelled in water, 6—8 kg. of sugar, and 1 kg. of swelled gelatin.

B. FULLMAN.

Production of complex aurothiosulphate compounds. E. and J. KEIDING (U.S.P. 1,640,775, 30.8.27. Appl., 3.5.26. Den., 7.11.25).—Aurous bromide or iodide dissolved in aqueous-alcoholic sodium bromide or iodide solution is treated with sodium thiosulphate to form sodium aurothiosulphate, which is precipitated by addition of alcohol. T. S. WHEELER.

Manufacture of solutions of bismuth salts of arylarsinic acids. A. HAYTHORNTHWAITE, and MAY & BAKER LTD. (E.P. 277,774, 16.7.26).—The insoluble bismuth arylarsinate is converted into a soluble double salt by addition of an alkaline base (ammonia, piperazine, etc.) and the solution is stabilised by means of an organic salt such as a tartrate or citrate; or the bismuth arylarsinate may be dissolved in tartaric or citric acid solutions with addition of the alkaline base. The bismuth arylarsinate may be formed *in situ*, *e.g.*, from a bismuthyl-tartrate or -citrate and a soluble arylarsinate, and the alkaline base and stabilising salt then added. A mixture of bismuth 3-acetamido-4-hydroxybenzenearsinate with 1 pt. of piperazine hydrate, 2 pts. of sodium potassium tartrate, and 5 pts. of water, nearly neutralised by addition of 0·35 pt. of 80% acetic acid, and made up to 100 pts. with water, gives a solution which can be sterilised at 110°. C. HOLLINS.

Manufacture of new formyl derivative of 2-[hydr]oxy-4-aminophenylarsinic acid [4-formamido-2-hydroxybenzenearsinic acid] and salts thereof. ÉTABL. POULENC FRÈRES, and E. FOURNEAU (E.P. 277,586, 7.6.27. Fr. 29.10.26).—4-Amino-2-hydroxybenzenearsinic acid is boiled with 98% formic acid for 3 hrs., giving *4-formamido-2-hydroxybenzenearsinic acid* (*sodium* and *ammonium* salts described).

C. HOLLINS.

Anti-spasmodics. F. Leuchs, Assr. to Winthrop Chemical Co., Inc. (U.S.P. 1,640,506, 30.8.27. Appl., 16.12.25. Ger., 24.3.25).—o-*Methylbenzyl carbamate*, m.p. 86°, p-*methylbenzyl carbamate*, m.p. 122°, and α-*phenyl*-n-*propyl carbamate*, m.p. 79°, are readily prepared by the usual methods. T. S. Wheeler.

Fixing agent for perfumes, volatile solvents, etc. J. D. Riedel A.-G. (G.P. 441,630, 21.1.26).—The material consists of the product obtained by the saponification with alkali hydroxides of wholly or partially hydrogenated or polymerised sperm oil or the like. L. A. Coles.

Manufacture of the active principle of lobelia. H. Wieland, Assr. to C. H. Boehringer Sohn Chem. Fabr. (Re-issue 16,772, 18.10.27, of U.S.P. 1,505,181, 19.8.24).—See B., 1925, 26.

Manufacture of calcium salts of inositol-phosphoric acid. A. Gams and M. Girard, Assrs. to Soc. Chem. Ind. in Basle (U.S.P. 1,645,233, 11.10.27. Appl., 9.4.23).—See E.P. 218,014; B., 1924, 731.

Manufacture of organic compounds (E.P.277,394).—See III.

***cyclo*Trimethylenearylpyrazolones** (E.P. 260,577 and 263,773).—See IV.

Acetic acid mother liquors (E.P. 266,684).—See V.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Action of potassium permanganate on the photographic plate, and exceptions to the law of reciprocity. J. Hrdlička (Compt. rend., 1927, **185**, 711—713).—An acid solution of potassium permanganate (0·2%) has been used to remove the sensitised grains of silver from an exposed and developed photographic plate. The rate of dissolution varied with the density of the plate. Treatment with the solution before exposure and development produced a decrease in the sensitiveness of the plate and in gamma, but pre-development tended to neutralise its effect. Treatment between exposure and development completely destroyed the latent image. A neutral solution, however, enabled those grains to be developed which had been rendered non-developable by the initial exposure. The acid solution is considered to destroy the silver sulphide "germs" (Sheppard), the latent image being formed partly from metallic silver. For the emulsion studied, gamma decreased with an increase in the illumination. J. Grant.

Mathematical methods of frequency analysis of size of particles. II. Application to silver bromide precipitates. R. P. Loveland and A. P. H. Trivelli (J. Franklin Inst., 1927, **204**, 377—389).—Thirty-three precipitates of silver bromide have been prepared which varied from each other only in two carefully-controlled factors. Size-frequency determinations of single layer plates made from these precipitates were made and analysed by the graphical method previously described (cf. B., 1927, 688). From these determinations it is concluded that in a chemical precipitation of silver halide the whole distribution of particles is not governed by a single-frequency law. W. E. Downey.

Colour of light sources and reflecting power of fabric. Cunliffe and Farrow.—See V.

Patents.

Manufacture of photographic silver halide emulsions. I. G. Farbenind. A.-G., Assees. of A.-G. f. Anilin-Fabr. (E.P. 259,926, 16.8.26. Ger., 14.10.25. Addn. to E.P. 258,237; B., 1927, 861).—Decomposition products of animal or vegetable proteins, prepared by acid hydrolysis, are added to a silver halide emulsion or to its constituents in order to bring about an increase in sensitiveness. Sensitiveness can be increased 30 times by adding a solution of products obtained by hydrolysing gelatin or other protein with about 1% nitric acid, the dissolved matter in the solution amounting to only about 2·5% of the amount of gelatin in the emulsion. W. Clark.

Protective layers for photographic films. I. G. Farbenind. A.-G. (E.P. 270,347, 2.5.27. Ger., 1.5.26).—Cellulose ether esters or mixed esters are used for the production of protective layers on the backs of photographic films. Cellulose acetate phosphate, applied in solution in β-methoxyethyl acetate, protects against electric discharges; ethylcellulose stearate, applied in alcohol–benzene–acetone solution, diminishes sliding friction in cinematograph films; cellulose acetate nitrate (2—3% N), applied in alcohol-benzene-acetone solution with a little monoacetin, prevents accumulation of electrostatic charges. C. Hollins.

Manufacture of a shadow-producing agent. B. Rapp (U.S.P. 1,644,446, 4.10.27. Appl., 17.5.26. Ger., 19.1.25).—Excess of moisture is removed from freshly-precipitated barium sulphate, and finely-divided sugar is mixed intimately therewith, the mixture is then spread in a thin layer, dried at 70—100°, and finely powdered by passing through a sieve. Such a preparation may consist of 100 pts. of barium sulphate, 12·5 pts. of cane sugar, and gum tragacanth. W. G. Carey.

Photographic copying by the reflection method. A. Miller, Assr. to I. G. Farbenind. A.-G. (U.S.P. 1,645,112, 11.10.27. Appl., 26.3.26. Ger., 14.11.24).—See E.P. 243,023; B., 1926, 997.

XXII.—EXPLOSIVES; MATCHES.

Sensitivities of detonating compounds to frictional impact, impact, and heat. C. A. Taylor and W. H. Rinkenbach (J. Franklin Inst., 1927, **204**, 369—376).—The frictional impact necessary to detonate various compounds has been determined by means of a pendulum friction machine. The frictional impact, impact, and heat necessary to explode twenty various detonating compounds have been determined. The results show that there does not exist a direct relationship between the values for different substances by any one test. It is concluded, however, that sensitivity

is not specific towards any one form of physical influence. W. E. DOWNEY.

PATENTS.

Acceleration of gelatinisation of cellulose nitrate. R. C. MORAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,640,712, 30.8.27. Appl., 19.6.24).—Gelatinisation of nitrocellulose by nitroglycerin is accelerated by addition of a small proportion of an aliphatic alcohol or glycol. T. S. WHEELER.

Explosive. P. R. DE WILDE, and SOC. SUISSE DES EXPLOSIFS (Swiss P. 117,568, 12.9.25).—An explosive, in the form of an oil (which can be used for the preparation of dynamite etc.), is obtained by the nitration of a mixture of glycerol and ethylene glycol. B. FULLMAN.

Safety explosives. A. B. M. GUILHON (F.P. 612,790, 15.3.26).—To chlorate explosives is added a polypicrate, obtained by suitable combination of picric acid with the carbonates of lead, calcium, copper, and magnesium. B. FULLMAN.

Explosives containing hygroscopic materials. E. BESSON (F.P. 617,285, 23.10.25).—The particles of the explosive are covered with a thin layer of a dry, pulverulent or colloidal mineral substance, *e.g.*, kaolin, waterglass, etc. B. FULLMAN.

XXIII.—SANITATION; WATER PURIFICATION.

Dissolved oxygen absorption test. II. E. A. COOPER and W. H. READ (J.S.C.I., 1927, **46**, 413—414 T; cf. B., 1927, 381).—The presence of small amounts of sodium permolybdate considerably increases the rate of dissolved oxygen absorption by sewage effluents. The oxygen absorption in two days in presence of the permolybdate tends to be of the same order as that observed in five days in absence of this catalyst. The preliminary results suggest the possibility of completing the Winkler dissolved oxygen absorption test in 48 hrs., instead of 5 days.

Water softening as practised at Oberlin, Ohio. W. H. CHAPIN (Ind. Eng. Chem., 1927, **19**, 1182—1187).—The water is softened by the lime-soda process, and methods of calculation of the amounts of chemicals required are given and reproduced in the form of graphs. After-precipitation of colloidal suspensions or of supersaturated solutions caused considerable trouble in the sand filters and in the mains even after 18 hrs.' settlement and filtration. The trouble was somewhat lessened by the addition of slightly basic alum (2% excess of Al_2O_3) at the rate of 30 pts./million. Later it was found that storage for about 4 weeks renders the water stable and prevents incrustation of pipes etc. even without filtration, the time factor and not re-carbonation being responsible. Long storage is much more effective than sand filtration for preventing after-precipitation. W. G. CAREY.

Determination of sulphuric acid in drinking water by use of benzidine. F. RASCHIG (Z. angew. Chem., 1927, **40**, 864).—Haase (B., 1927, 798) is mistaken in condemning Raschig's method (A., 1906, ii, 306) for determining sulphuric acid in such small proportions as are present in drinking water. Provided the prescription is exactly followed (the benzidine solution should contain 40 g. of benzidine per litre) and the quantity of water taken is adjusted to the sulphuric acid present, the results obtained are accurate. W. A. SILVESTER.

PATENTS.

Production of disinfecting agents. A. BOEHRINGER (C. H. BOEHRINGER SOHN) (E.P. 253,918, 16.6.26. Ger., 20.6.25).—Lactates or alkalis are added to lactic acid until the p_H value is between 3·25 and 4·2, or lactates are treated with acid, *e.g.*, sulphuric acid, to give the requisite hydrogen-ion concentration. Nutrient media such as peptone or carbohydrates, odoriferous material such as pine oil, or cultures of lactic acid bacteria may be added. W. G. CAREY.

Volumetric displacement apparatus for controlling the supply of gas for chlorinating water etc. B. BRAMWELL (E.P. 277,869, 16.3.27).—A displacement vessel closed at the top and open at the bottom is contained in a closed water vessel and carries a U-shaped vent tube, one end of which projects outside the vessel, the other end being enlarged to give equal and positive displacements of gas without bubbling. The U-tube is so disposed that the bottom is above the lower edge of the displacement vessel. W. G. CAREY.

Prevention of boiler-scale formation. LA SUVAPO (SOC. ANON.) (F.P. 617,870, 6.11.25).—To the boiler feed-water are added a cerium salt susceptible to oxidation, tannin, and an alkali, *e.g.*, a mixture of 3% of cerous sulphate, 37% of tannin extract (d 1·2), and 60% of sodium carbonate. B. FULLMAN.

Method of diminishing the internal rusting of hot-water apparatus. C. HÜLSMEYER (Swiss P. 117,715, 7.7.25).—The entrapment of air in the feed water to boilers and other hot-water apparatus is rendered harmless by passing the water through a vessel packed with manganese steel wool, which removes the dissolved oxygen. A. R. POWELL.

Softening of water by base exchange. K. MORAWE (Swiss P. 117,570, 26.2.25. Ger., 25.3.24).—The water is treated with a moulded, alkaline, base-exchange softening material capable of regeneration. The treatment is such that the water only acts on the outer layers of the pieces, which may then be regenerated in a shortened time. B. FULLMAN.

Utilisation of zeolites [for water-softening]. W. H. GREEN, Assr. to GENERAL ZEOLITE Co. (U.S.P. 1,644,469, 4.10.27. Appl., 8.6.23).—Hard water is passed continuously upwards through a portion of a bed of zeolite, and a regenerating solution is passed intermittently downwards through another portion of the zeolite, and is washed out, after which particles of the bed are transferred from each portion to the other. W. G. CAREY.

Impregnating compounds (E.P. 277,083).—See XIII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DECEMBER 9, 1927.

I.—GENERAL; PLANT; MACHINERY.

Producing small bubbles of gas in liquids by submerged orifices. C. G. Maier (U.S. Bur. Mines, Bull. 260, 1927, 62—120).—Regulation of the dimensions of gas bubbles is necessary for the successful catalytic autoxidation of ferrous sulphate and sulphur dioxide. For a static bubble of low gas velocity ruptured only by buoyancy, in water at 20° with perfect wetting of the tip, $r = 9 \cdot 05 R^3$, where R is the radius of the free bubble and r that of the tip, both in cm. When there is a finite gas velocity R will also be modified by the effect of circulation and viscosity. The size of bubbles was determined in practice by counting, by the bubble burette (a tapered burette in which one bubble was caught), by photography, and by conductivity determinations. The last method depends on the fact that the resistance of a column of liquid up which a number of uniform bubbles are rising depends on the total gas volume and the average bubble size. For accurate results this method requires that the bubble size should not be too small relative to the column. By these methods the size of bubbles produced by various tips was determined. The results showed that the size was greatly influenced by gas pressure or velocity in the tip. Small bubbles produced by simple tips require very small orifices and in consequence uneconomically high pressures. The internal diameter of the tip only is of importance if wetting is perfect, so a perforated plate should give similar results to a single tip. Results with static bubbles were in good agreement with calculation. With increase in gas velocity the size passed through a minimum. The prime factor in selecting a material for the orifice is that it shall be perfectly wet. For bubbles below 1 mm. in diameter the pressure required to overcome surface tension is such that the power required would be excessive. Agitation devices for breaking up bubbles are not susceptible of accurate control. The size of bubbles can, however, be reduced by giving the liquid a motion at right angles to the bubble tip. In this way the grid type " shear bubbler " was designed. It consists of a grid of parallel pipes spaced closely with orifices near the plane of the axes of the pipes. The dimensions of such a bubbler for a given output are calculated, and also the power required for pumping liquid. Either a grid of lead or a fabric diaphragm (*e.g.*, woollen filter cloth) is thought most likely to be suitable for the process under investigation.
C. Irwin.

Automatic apparatus for the determination of moisture [by distillation with benzol etc.]. H. Rössler (Chem.-Ztg., 1927, **51**, 688—689).—A wide-necked Erlenmeyer flask with ground-glass stopper is connected by the latter to a measuring cylinder holding benzol or the like and graduated in 50 c.c. A side-tube from a bulb above the stopper connects to the base of a reflux condenser. Below this is a tube with run-off tap graduated in 1/10 c.c. in which the distilled water is collected. The flask is intended particularly for the ready weighing of pasty liquids. C. Irwin.

Boiler-scale prevention. Sauer and Fischler.—See XXIII.

Patents.

Pulverisers. F. L. Duffield (E.P. 277,734, 15.6.26). —A disintegrator is combined with a preliminary crusher; the latter has yielding parts to permit the passage of uncrushable material. The first set of beaters of the disintegrator serve also as fan, and an additional fan is placed after the beaters, but mounted on the same shaft, to draw material and air through the apparatus.
B. M. Venables.

Ball mills. E. C. R. Marks. From Traylor Engineering & Manuf. Co. (E.P. 278,214, 6.12.26).—Transverse screens for tube or ball mills are constructed of a number of grate bars, all individually alike and easily renewable, which are held in a frame so that the resulting screen is conical with the base towards the inlet. Beyond the screen is a diaphragm plate with central hole, and between the diaphragm and screen are lifters to pass the undersize into the next grinding compartment or to the final discharge. B. M. Venables.

Crushing and mixing mills of the edge-runner type. H. Martinez and R. H. Kirk (E.P. 278,236, 8.2.27).—The edge-runners have non-radial axes and are provided with vertical adjustment either together or individually and with means for applying spring pressure. B. M. Venables.

Edge-runner mills. R. B. Lucas (E.P. 278,069, 28.6.26).—In an edge-runner mill a number of discs are grouped on a shaft which is offset to the vertical driving shaft, *i.e.*, the runner shaft is not radial. The discs may themselves be made heavy or pressure may be applied by an additional weight or spring. A runner of normal type may or may not be used in addition.
B. M. Venables.

Disintegrating machine. D. C. Addicks (U.S.P. 1,643,938, 4.10.27. Appl., 3.4.26).—A crushing chamber with a bottom grating is mounted with a feed hopper in a housing, and a rotor in the chamber co-operates with a breaker plate. The material is fed into the chamber by a gyrating plate located between the breaker plate and a pendant hinged plate forming a movable bottom for the hopper. The gyrating plate is driven

from the rotor, and its lower end reciprocates on the breaker plate and slides over the movable bottom.
H. HOLMES.

Separation of solid materials from meal or powder. A. W. SIZER (E.P. 277,817, 9.10.26).—The material is delivered to a shaking screen or table provided with upstanding pins, preferably in staggered relation, and at its discharge end with a lateral trough, preferably pivoted, of which the discharge lip is adjustable in height. Materials, *e.g.*, string, wood, etc., not engaged by the pins are retained by the trough.
H. HOLMES.

Suction and filter apparatus for removing dust from grinding and like machines. F. H. and H. S. POCHIN (E.P. 278,156, 6.9.26).—A dust-collecting fan with individual inlet ducts from each source of dust and fabric-collecting filters. B. M. VENABLES.

Mixing apparatus. A. B. and C. R. SMITH (E.P. 278,177, 5.10.26).—In addition to moving blades for throwing the material about, the mixer has fixed blades or inclined pressure surfaces which subject the material to a squeezing or wedging action. B. M. VENABLES.

Concentrator table. A. H. STEBBINS (U.S.P. 1,644,753, 11.10.27. Appl., 10.11.24).—A deck mounted for reciprocation in a supporting frame is provided with a surface comprising unapertured sections with intervening sections having apertures through which air under pressure is supplied from below. The materials delivered to the deck are thus subjected alternately to the grading action of the deck alone, and to that of the air and moving deck combined. The concentrates on reaching an apertured section fall through against the lift of the issuing air. Lips at the apertures prevent the entry of the tailings. H. HOLMES.

Method of drying material. J. E. ALEXANDER (U.S.P. 1,645,366, 11.10.27. Appl., 28.11.24).—Variable degrees of heat concentration are applied to the material in the drying chamber, while maintaining a uniform temperature of the superheated steam in the chamber surrounding the material. H. HOLMES.

Dryer or dehydrating plant. F. F. KNIPSCHILD (U.S.P. 1,645,760, 18.10.27. Appl., 10.5.26).—A heat-circulating system within the drying enclosure is provided with an outlet and with guide means enabling it to draw in fresh air from the space immediately around the enclosure. H. HOLMES.

Spray drying. INDUSTRIAL ASSOCIATES INC. (E.P. 278,263, 4.4.27. Addn. to E.P. 272,859; B., 1927, 801).—In the apparatus described in the original patent several nebulisers or atomisers are used all at about the same level. Since each central spray is surrounded by its own downwardly-flowing stream of hot air there is no danger of mist from adjoining sprayers coalescing into drops. B. M. VENABLES.

Apparatus for atomising liquids. S. WRIGHT (E.P. 277,912, 9.6.27).—Liquid is lifted by helical vanes on the interior of an inverted cone, which is in rapid rotation. The liquid passes through openings in the cone over the surface of discs attached to its exterior. A number of spraying points are formed on the edges of the discs by notching and twisting them, and the discs are surrounded by notched strips preferably arranged helically. B. M. VENABLES.

Separation of solids from liquids. S. SYMINGTON (U.S.P. 1,641,708, 6.9.27. Appl., 26.6.20).—The mixture of solid and liquid is delivered on to an endless fabric belt which is mechanically folded to enclose the mixture and passed over a suction box to remove the liquid. The belt is next led through a stream of washing liquid, which is removed by a second suction box, and after passage through a dryer the belt is mechanically unfolded and the solid removed from it. T. S. WHEELER.

Evaporators. H. HILLIER (E.P. [A] 278,075 and [B] 278,299, 29.6.26).—(A) The evaporator comprises a shell containing the liquid to be evaporated and a space for vapour. Projecting from the side of the main shell is another shell for heating medium, within which is a bank of horizontal or inclined tubes opening at one end to the liquor space in the main shell and at the other to a header within the projecting shell. To allow for expansion the header is not attached to the small shell, but only to the tubes. The tubes in the lower row or rows are larger in diameter and serve for the inward flow of the circulation. (B) The upper tubes which serve for the outward flow of liquid plus vapour increase in length downwards so as to obtain approximately equal flow through each; to effect this the tube plate within the main shell is stepped or inclined. Guide plates are placed within the main shell to deflect the issuing liquor and vapour towards the surface of the liquor.
B. M. VENABLES.

Crystallisation of liquids. G. T. WALKER (U.S.P. 1,644,161, 4.10.27. Appl., 24.3.24. Cf. U.S.P. 1,615,151; B., 1927, 176).—The hot solution is progressively cooled as it passes through a container where its level is maintained constant with overflow discharge. Crystallisation is assisted by devices which lift the crystals and drop them through the solution. The crystals are advanced through the container and finally lifted for discharge with the solution. H. HOLMES.

Fluid filters. SVENSKA ACKUMULATOR AKTIEBOLAGET JUNGNER, Assees. of B. H. LUNDBORG (E.P. 272,841, 9.11.26. Swed., 19.6.26).—The filter is of the type where the filtrate passes between the spaces left between an inner cylinder and a surrounding sleeve, each being formed with grooves, the valleys on one being opposite the hills on the other. The cylinder and sleeve are tapered so that the size of the interstices may be adjusted by relative longitudinal movement. B. M. VENABLES.

Apparatus for filtering. W. H. FURNESS, Assr. to CELLOCILK Co. (U.S.P. 1,643,299, 27.9.27. Appl., 6.12.24).—The filter is composed of a casing made up of a lower and an upper casting, the former being provided with an inlet and the latter with an outlet tube. The filtering element, which is secured to the upper casting, consists of a number of plates the lower surfaces of which are entirely flat, but with upper surfaces consisting of a flat central portion surrounded by an outer portion which tapers towards the lower surface from a point slightly below the level of the flat central portion. The liquid is enabled to pass through the plates by means of holes adjacent to the central flat portion, communication being established between these holes

by means of a channel or groove, whilst the solid material is held back at various points in the taper between the plates according to the size of the particles.

F. R. Ennos.

Decolorisation of aqueous liquids. Brit. Dyestuffs Corp., Ltd., C. Hollins, and E. Chapman (E.P. 278,485, 24.8.26).—Certain wetting-out agents, namely alkylnapththalenesulphonic acids, ligninsulphonic acid, naphthenic acids, sulphonated *iso*propylated mineral oil fractions, and sulphonated higher fatty acids, facilitate contact between the adsorbent substance (charcoal, silica, etc.) and the liquid to be decolorised, and thus accelerate decolorisation. The substances are added either to the liquid or to the adsorbent material.

C. Hollins.

Washing apparatus involving the intermixing of gases and liquid. E. L. Pease (E.P. 278,105,7.7.26). —Liquid is circulated between upper and lower chambers by a chain pump. In the rising pipe the discs fit closely and lift the liquid, which descends in the other pipe, where there is considerable clearance, and through which the gas passes con- or counter-current to the falling liquid. A number of units may be used in series for counter-current washing. B. M. Venables.

Apparatus for cooling and purifying gases. Richardsons, Westgarth, & Co., Ltd., and F. G. Inglis (E.P. 278,118, 21.7.26).—On one or both sides of a fan are placed alternate fixed and rotating perforated discs, the latter being on the fan shaft. Jets of liquid are admitted to the spaces between the discs through holes in the boss driving the rotating discs. The gas passes axially through the discs, and is exhausted by the fan. B. M. Venables.

Filtration of gases with a high oxygen content. I. G. Farbenind. A.-G., Assees. of A. Benischek (G.P. 442,504, 22.9.25).—Liquid triaryl phosphates are used for removing dust from the gases. L. A. Coles.

Method and apparatus for [heat-]treating materials. Dwight & Lloyd Metallurgical Co., Assees. of R. W. Hyde (E.P. 269,480, 26.11.26. U.S., 16.4.26).—A number of grates or perforated pallets are attached to a chain conveyor which travels beneath a low roof of refractory material. Flame is produced in combustion chambers below the roof alongside the conveyor, and the hot gases are drawn down through the material (*e.g.*, alkaline-earth carborates, clay products, etc.) on the conveyor to wind (vacuum) boxes below. At the feed end provision is made for preheating the material by passing the gases through twice, and at the outlet end for continuing the reaction and cooling by passing the material under a continuation of the roof which is not provided with combustion chambers or wind box. B. M. Venables.

Muffle furnace. W. G. Bridge and W. A. Pervier, Assrs. to C. E. Parkhurst (U.S.P. 1,644,107, 4.10.27. Appl., 17.4.26).—A refractory lining within a layer of heat-insulating material is provided with means for heating it electrically. The lining comprises a series of plates, the series being removable from the muffle, and each plate readily separable from the series.

H. Holmes.

Annular rotary-hearth ovens. J. E. Pollak. From Trocknungs-, Verschwelungs-, u. Vergasungs-Ges.m.b.H. (E.P. 278,207, 23.11.26).—An annular hearth is constructed in three stories, connected together by hinge-like members to allow for distortion. The lower story comprises the carriage and driving mechanism below the heating zone, the middle comprises a non-conducting bed of the furnace and gas-tight joint, and the upper is a conducting hearth or platform which supports the goods and is arranged at a considerable height above the bed, so that the platform may be heated from below by radiation from the lower part of the furnace walls. Fixed walls and roof are provided enclosing the two upper hearths. B. M. Venables.

Furnace walls. Simon-Carves, Ltd., and B. F. Clark (E.P. 278,159, 9.9.26).—A furnace wall is provided with heat-insulation containing at least one air space, a mattress of solid heat-insulation is held against the inner wall (*e.g.*, the water wall of a boiler furnace) by means of round iron rods laid longitudinally in V-shaped spaces formed between the mattress and inclined lugs attached to the outer wall across the air space. Under expansion and contraction strains the iron rods can follow up or rise. B. M. Venables.

Heat-exchange apparatus. J. Muchka (E.P. 272,152, 16.7.26. Austr., 7.6.26).—A heat exchanger is constructed of flat rectangular plates joined together by U-shaped members at opposite edges, alternately the vertical and horizontal edges. Distance pieces in the form of bars are also inserted. B. M. Venables.

Heat-exchange apparatus. B. F. Sturtevant Co., Assees. of G. C. Derry (E.P. 254,702 and 269,560, 23.6.26. U.S., 6.7.25).—A heat exchanger, *e.g.*, for use as an economiser in a boiler flue, is constructed of a number of horizontal tubes, of which the heating surface is increased by flange-like fins, and in which the water zig-zags across and downwards with an upward flow of the flue gases. Only a few tubes are connected to any one header, and they are supported to allow for free expansion and contraction, and are easily cleaned or removed. B. M. Venables.

Sublimation apparatus. C. Field, Assr. to Nat. Aniline & Chemical Co., Inc. (U.S.P. 1,644,518, 4.10.27. Appl., 11.2.21).—Pulverulent material which becomes plastic at the sublimation temperature is subjected to continuous sublimation by the use of a receptacle provided with a heating jacket and with stirrers operating adjacent to the heated walls. A spraying device converts the highly heated residue into granules adapted for ready removal through a discharge opening. H. Holmes.

Process of refrigeration. W. H. Carrier, Assr. to Carrier Engineering Corp. (U.S.P. 1,642,942—3, 20.9.27. Appl., [a] 23.4.23; [b] 22.3.24).—(a) *s*-Dichloroethylene is suitable as a working fluid for refrigerating machines. (b) It is preferable to separate it by fractional distillation into its stereoisomerides, and to use one or other separately. T. S. Wheeler.

Fire-extinguishing compound. "Poleo" Feuerlösch Apparate G.m.b.H. (E.P. 273,713, 27.6.27. Ger., 30.6.26).—The compound consists of 60% of methyl bromide, 35% of carbon tetrachloride, and 5% of ethylene bromide. H. Royal-Dawson.

Friction surfaces etc. Brit. Dyestuffs Corp., Ltd., C. J. T. Cronshaw, J. Baddiley, and E. Chapman (E.P. 278,465, 21.7. and 3.11.26).—The slipping effect due to water on a braking surface is greatly diminished and the braking power may be increased by the presence at the braking surface of emulsifying, dispersing, or wetting-out agents, *e.g.*, *iso*propylnaphthalenesulphonic acids, sulphonated aldehyde–phenol condensation products, ligninsulphonic acid, sulphonated acid resins, etc., and in particular the sulphonated *iso*propylated petroleum fractions of E.P. 274,611 (B., 1927, 841). These substances similarly prevent the slipping of driving-belts on pulleys. They are applied ordinarily as aqueous solutions of their sodium salts. C. Hollins.

Continuously-operating apparatus for gas analysis. O. Rodhe, Assr. to Svenska Aktiebolaget Mono (U.S.P. 1,644,951, 11.10.27. Appl., 15.12.22. Swed., 15.12.21).—The gas mixture is passed through a first group of cells in which the percentage of carbon dioxide is determined, thence through means for oxidising carbon monoxide and methane to carbon dioxide, and finally through a second group of cells in which the percentage of carbon dioxide in the resultant mixture is determined. H. Holmes.

Instrument for measuring fluids for turbidity, colour, and other characteristics. W. G. Exton (U.S.P. 1,644,331, 4.10.27. Appl., 23.10.25).—The fluid is contained in a transparent receptacle supported within a casing provided at one end with an observation opening and open at the other end to admit light. A target member is mounted in this open end, and a measuring wedge is slidable transversely through the casing between the eye-piece and the sample under test. H. Holmes.

Apparatus for determining the specific gravity [of gases]. G. B. Linderman, jun., Assr. to Amer. Meter Co. (U.S.P. 1,644,684, 11.10.27. Appl., 27.5.24). —Two pressure-developing centrifugal devices, operated at a predetermined speed ratio, are supplied the one with air and the other with the gas to be tested, and means are provided for maintaining equilibrium between the pressures of the air and gas supplies. The outlet from each device is connected to one limb of a corresponding manometer, the other limb of which is subjected to the pressure of the air and gas supplies and is provided with a float. The floats operate means for indicating the relative sp. gr. of the gas and air. H. Holmes.

Separation of two or more substances from a solution. I. Isaachsen, Assr. to A./S. Krystal (U.S.P. 1,646,454, 25.10.27. Appl., 26.2.23. Norw., 29.3.22).—See G.P. 392,193; B., 1924, 584.

Continuous determination of a constituent of a mixture of gases. R. P. Mase (E.P. 271,027, 8.11.26. U.S., 15.5.26).—See U.S.P. 1,634,331; B., 1927, 592.

Refrigerating plant. I. Amundsen (U.S.P. 1,647,208, 1.11.27. Appl., 29.10.26. Norw., 28.1.26).—See E.P. 266,683; B., 1927, 592.

[Blocks for] furnaces having suspended arches. L. Morton and J. Hargrove (E.P. 277,768, 7.7.26).

[Slabs for] heat insulation. R. W. Anderson (E.P. 277,747, 22.6.26).

Separation of solids from liquids (E.P. 277,500).—See IX.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Chemical constituents of lignites. J. Marcusson (Z. angew. Chem., 1927, 40, 1104—1106).—Lignites contain, on an average, about 69% C, 5·5% H, 25% O, and 0·8% N (reckoned on the ash-free, dry substance). The constituents, other than water and mineral matter, are waxes, resins, free humic acids, humic acid anhydrides, humic ketones, water-soluble carbonyl acids, acetic acid, lignin, and cellulose. Waxes and resins are extracted with carbon tetrachloride, and separated with a mixture of alcohol and ether at a reduced temperature, the waxes being insoluble. Free humic acids are removed with ammonia, and humic acid anhydrides are hydrated with hot 1% sodium hydroxide. Lignin and ketones are separated by mixing with molten resorcinol, the ketones remaining unchanged. Cellulose is determined by chlorination. Lignites derived from tree trunks are principally composed of humic acid anhydrides, lignin, cellulose, and water-soluble carbonyl acids. In earthy lignites, lignin and cellulose have been almost completely converted into humic acids and anhydrides. The content of humic ketones increases with the age of the lignite. Alkali-soluble lignites contain a high proportion of free humic acids, and their cellulose is in the form of oxycellulose, so that they are almost completely soluble in cold 1% sodium carbonate. Water-soluble carbonyl acids present in lignites have reducing properties and comprise a mixture of glycuronic acid and an acid derived from it by oxidation. W. T. K. Braunholtz.

Mechanical strength of coke. G. Dörflinger (Stahl u. Eisen, 1927, 47, 1867—1871).—The strength is determined by placing 50 kg. of the coke in pieces of about 50 mm. in diameter in a rotating cylinder which is rotated through 100 revolutions in 4 min. The coke is then screened on a series of screens with holes from 40 mm. to 10 mm. At least 72% should remain on the 40 mm. screen and only a very small quantity of material finer than 10 mm. should be obtained.
A. R. Powell.

Chemical methods of treating wood: use of waste material and wood preservation. C. Schwalbe (Z. angew. Chem., 1927, 40, 1172—1176).—A general survey of the application of the products of German forestry. The life of the wood-distillation industry may be extended if the slowness and low thermal efficiency of the usual distillation at 500° can be avoided. The temperature required may be lowered by the use of high pressures, or by the use of a solution of magnesium or calcium chloride with strong sulphuric acid. In the latter case a temperature of 180° and a pressure of 5·6 atm. is sufficient. The hydrochloric acid liberated hydrolyses the cellulose to sugar which is carbonised by the sulphuric acid. The yield of methyl alcohol and acetic acid is greatly increased, no tar being produced. The charcoal is of lower calorific value than ordinary wood charcoal, but may be given a second dry distillation if desired. Both this process and the proposed hydrolysis to sugar have the disadvantage that they usually involve large central plants with carriage charges to them for sawdust etc. that could scarcely be borne. The bulk of waste product discharged from sulphite-pulp mills is very large. Its only likely large-scale application is as

fuel. It can be carbonised at high pressures, but this is too costly. Addition of adsorption carbon, which can be cheaply made by the low-temperature carbonisation of shavings (*vide supra*), to the waste renders it possible to carbonise it completely by heating at 10 atm. A yield of 60—70% of charcoal is obtained, with wood alcohol and acetic acid as by-products. This process also involves certain practical difficulties. Neither does the manufacture of hemicellulose offer an outlet for waste wood products. A chemical method of removing bark in sulphite-pulp manufacture would give an appreciable saving in material. If undried wood could be impregnated, *e.g.*, with sodium fluoride, as some experiments suggest, it might be possible to sterilise and market trees felled in consequence of disease. C. Irwin.

Distillation of benzol wash-oil under diminished pressure. F. Raschig (Z. angew. Chem., 1927, **40**, 1089—1090).—Ordinary benzolised oil is distilled under diminished pressure at about 140°, without the use of free steam, in a still 10 m. high. The vapours pass through a condenser in which they give up their heat to the inflowing rich oil, so that the latter enters the still at 120—130°. The quantities of steam and cooling water required are, respectively, three times and forty times the quantity of benzol made, as against six times and a hundred times in the usual process. Thickening of the wash oil is much less pronounced, and the benzol vapours are free from water and can be fractionally condensed. To avoid any benzol losses due to incomplete condensation under diminished pressure, the lightest fraction is taken off and condensed under only moderately reduced pressure, the heavier fractions being subsequently distilled under a higher vacuum. W. T. K. Braunholtz.

Esthonian oil shale and its economic importance. I, II. P. W. Uhlmann (Chem.-Ztg., 1927, **51**, 745—746, 766—767).—I. The Esthonian oil shale deposit covers an area of about 3000 km.^{2} on the Russian-Esthonian border, and the amount of material available is estimated at 7·2 milliard tons. Analysis of various samples showed the content of organic matter to vary from 35·7 to 51·7%. The air-dried shale consisted of volatile matter 42·7—60·1%, coke 2·9—9·7%, and ash 36—53%. The organic matter contained 71·5—72·4% C, 8·4—8·7% H, 18·7—20·1% O, and 0·2—0·3% N, but no sulphur. Any sulphur present in the shale itself (0·4—1·5%) must therefore be in combination with the inorganic material. Various industrial applications of the shale are indicated, amongst which are the utilisation of its distillation products in the manufacture of asphalt, and of the inorganic material present in it and containing silica, alumina, and lime, for use in the cement industry.

II. The shale is distilled in shaft-like retorts and heated by a current of oxygen-free gas from a gas producer. The gas, produced at 600—1000°, is combined with the cooled gas from the still to reduce the temperature to about 500°, at which temperature the distillation takes place. Six such stills have a throughput of 200 tons of shale per day, producing 30—40 tons of oil. One ton of shale produces about 280 cub. m. of gas, having a calorific value of 4300—4500 kg.-cal./cub. m. and the following composition: 37·6% H_2, 25·8% CH_4, 1·6% C_2H_4, 19·1% CO, 13·3% CO_2, and 2·6% N_2. Tar (3·5%) and a large proportion of aromatic hydrocarbons as well as phenols are produced. The amount of shale oil produced (16—25%) depends on the method of preparation. From the washing oil in the gas washer is recovered a benzine (*d* 0·76) with a volatility of 29% at 100°. A second fraction, resembling petroleum, has *d* 0·813—0·850, flash point 36°, and contains 0·65—0·77% S. A third fraction also occurs having *d* 0·900, flash point 55—60°, and containing 0·62% S. Phenols occur in the fraction 180—360° in proportions ranging from 20 to 50%, depending on the temperature of distillation of the shale. The use of superheated steam also raises the proportion of phenols. The residue remaining after the distillation of the crude oil above 360° contained pitch and asphalt. Crude shale oil consists of neutral substances (chiefly hydrocarbons) 72·1%, higher phenols 22·4%, carboxylic acids 4%, and basic substances 0·2%. It has *d* 1·004, flash point (P.-M.) 84°, unsaturated compounds 81%, and viscosity at 50° (Engler) 5·38 (cf. Kogerman, B., 1927, 865). R. C. Odams.

Influence of admixture of salts and certain organic compounds on the oxidation of highly refined [transformer] oils. H. von der Heyden and K. Typke (Petroleum, 1927, **33**, 1253—1254).—150 g. of the oil were mixed with 0·5 g. of each of the various compounds in turn and heated for 7 hrs. at 120° in a flask through which a current of oxygen was passed. After oxidation the acid value, sulphuric acid-soluble material, and tar and saponification values were determined. The solubility of the oil in sulphuric acid was found to be a good indication of the change produced, as in a non-sludging oil this factor did not increase after oxidation. Of the substances used, magnesium oxide and carbonate gave somewhat low tar and acid values. The addition of caustic soda or of sodium or potassium carbonate accelerated the oxidation; ferric oxide had an extraordinarily unfavourable influence. Again, lead monoxide checked the oxidation of the oil, whilst red lead oxide (Pb_3O_4) gave an acid value about the same as that obtained by oxidation of the oil alone, the saponification value, however, being increased considerably, and lead dioxide greatly increased the oxidation. Of organic compounds, phenol, starch, oxalic and oleic acids accelerated oxidation, whilst salicylic acid, hydroxylamine hydrochloride, and especially pyrogallol and aniline had the opposite effect. These results are in contradiction to those of Butkov (B., 1926, 1002), who found that both phenol and aniline were without action on the oil.

R. C. Odams.

Shale oil as wood preservative. Sowder.—See IX.

Patents.

Treatment of oil and coal and production of liquid hydrocarbons therefrom. F. Lamplough and A. E. Hodgson (E.P. 277,419, 15.6. and 11.8.26).—From 10 to 50% by wt. of finely-divided coal is mixed with a heavy mineral oil in such a manner that a stable dispersion of coal in oil is formed. The suspension so formed is subjected to high temperature (500—600°) and pressure (100—600 lb./in.^{2}), and the liquid fractionally distilled. The oil is apparently hydrogenated by the coal, and the process may be furthered to such an extent that the coal, with the exception of the ash, goes almost

completely into the liquid state. The heaviest distilled fractions may be used as a binder in the manufacture of briquettes. Fractional condensation of the oil vapours may be effected in apparatus as described in E.P. 216,922 (B., 1924, 665). The distillation vapours may be hydrogenated and redistilled for spirit. R. C. ODAMS.

Motor fuels. E. V. BERESLAVSKY (E.P. 258,550, 30.9.26. U.S., 17.9.25).—Low-compression fuels, such as gasoline or kerosene, are adapted for use in high-compression engines without "knocking" by the addition of mesitylene, its amino-compounds, or their derivatives. The mesitylene may be dissolved in alcohol or some other blending agent and so admixed with the fuel, or it may be atomised in the engine intake, or fed into the combustion chamber before ignition. R. C. ODAMS.

Utilisation of sludge fuel. GES. FÜR MASCHINELLE DRUCKENTWÄSSERUNG, M.B.H. (G.P. 441,669, 5.5.25).—The fuel is briquetted without binding medium, the briquetting plant being placed near the furnace and the briquettes being delivered directly to the furnace chamber so as to avoid crumbling. C. HOLLINS.

Prevention of [coal] mine explosions. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,642,912, 20.9.27. Appl., 20.10.24; cf. U.S.P. 1,479,757; B., 1924, 208).—The walls of the passages of the mine are sprayed with a high-boiling oil with a high flash-point. T. S. WHEELER.

Manufacture of oil gas. R. D. PIKE (U.S.P. 1,644,146, 4.10.27. Appl., 2.5.23. Renewed 28.2.27).—Oil previously treated with steam is passed over heated surfaces in a closed chamber, whereby the oil is converted into a lean gas carrying particles of suspended carbon. The gas is treated in another closed chamber with steam at a predetermined temperature, and the carbon is thus converted into gas. The lean gas is subjected to further treatment and thereby enriched. R. C. ODAMS.

Process and apparatus for cracking hydrocarbons. H. J. JANSEN, JUN. (E.P. 278,235, 7.2.27).—Hydrocarbons are cracked by forcing them in a horizontal direction through a vessel containing a metal (lead) and/or metallic salt (zinc chloride) in a molten condition. The vapour outlet has only a small free surface, so that carbon deposit is removed in suspension by the vapours, which travel at a high velocity. Heavy liquid residue is immediately run off the surface of the molten material. R. C. ODAMS.

Apparatus for cracking oils. O. KAY (E.P. 276,947, 8.1.27. U.S., 4.9.26).—Oil is introduced into an atomiser consisting of a frusto-conical nozzle rotating in a housing open at one end so that the oil impinges radially upon the inner walls of the nozzle. Air is introduced axially into the cone, and cracking takes place by the bombardment of the inner walls of the cone by the hydrocarbon molecules. The various products are separated by causing portions of the spray of varying densities to fall into vessels suitably placed. R. C. ODAMS.

Treatment [cracking] of hydrocarbons. R. DE M. TAVEAU, Assr. to TEXAS CO. (U.S.P. 1,645,553, 18.10.27. Appl., 16.3.23).—The cracking process consists in vaporising the oil in a still so constructed that it contains a quiescent pool of aluminium chloride which is not in contact with the heated bottom of the still. R. C. ODAMS.

Treatment [cracking] of hydrocarbon oils. R. C. HOLMES, F. T. MANLEY, and O. BEHIMER, Assrs. to TEXAS CO. (U.S.P. 1,646,380. 18.10.27. Appl., 10.12.23).—Oil is heated to cracking temperature and passed into a number of converters connected in series and maintained at that temperature. A portion of the generated vapours is condensed and refluxed back to the several converters. R. C. ODAMS.

Continuous distillation of hydrocarbon oils. J. R. CARRINGER, Assr. to STANDARD DEVELOPMENT CO. (U.S.P. 1,644,324, 4.10.27. Appl., 6.5.21).—In a battery of continuous stills, each having a fractional condenser, a common pressure-equalising chamber is fitted, together with means for connecting the vapour spaces of the condensers to the equalising chamber. R. C. ODAMS.

Decomposition of gaseous or vaporous hydrocarbons by means of water vapour. I. G. FARBENIND. A.-G. (E.P. 265,989, 11.2.27. Ger., 11.2.26).—A mixture of hydrocarbons and water vapour is decomposed by the action of heat into hydrogen and carbon monoxide. The highly heated gases obtained from the decomposition are cooled by contact with cold water. The water thus heated is brought into direct contact with fresh gases containing hydrocarbons to be subjected to the reaction, thus heating the gases and charging them with water vapour. The resulting gas mixture is then introduced into the decomposition chamber, where carbon monoxide and hydrogen are produced. If pure hydrogen is desired, the gas issuing from the decomposition chamber is passed, with further additions of steam or water, over a suitable catalyst, preferably at a lower temperature, and the carbon monoxide and carbon dioxide are eliminated. R. C. ODAMS.

Preparation of alcohols from olefine-bearing gases. PETROLEUM CHEMICAL CORP., Assees. of H. S. DAVIS (E.P. 249,834, 1.3.26. U.S., 30.3.25).—The mixed gases containing olefines, especially gases from cracked oils, are freed from hydrogen sulphide by means of iron oxide and scrubbed with 80% sulphuric acid to remove water and olefines more reactive than propylene and ethylene. The propylene is then absorbed in 100% sulphuric acid at 1—2 atm. pressure in towers cooled below 30°. The product is diluted with water and the constant-boiling mixture of *iso*propyl alcohol and water is distilled off. A preferred method uses six towers, two for 80% sulphuric acid and four for 100% sulphuric acid; the towers are 24 ft. high and 6 in. diameter, the gas flow being 10 cub. ft. per min., and the 100% acid flow 2—3 gals. per min. Dilution of the acid by the absorption products may be compensated by addition of oleum. The use of 100% acid enables 1 mol. of acid to absorb as much as 2 mols. of propylene, and these concentrated absorption products are much more stable than those obtained with 90% acid. C. HOLLINS.

Manufacture of a clarifying and decolorising agent [for oils]. M. L. CHAPPELL, R. F. DAVIS, and M. L. MOORE, Assrs. to CONTACT FILTRATION CO. (U.S.P. 1,642,871, 20.9.27. Appl., 1.8.21).—Bentonite clay mixed with water is heated with sulphuric acid at 100°

for 14 hrs., the mixture being thoroughly agitated by air. The solid residue is washed with water and dried.
T. S. Wheeler.

Separation of oily emulsions. G. W. Coggeshall and A. Reilly, Assrs. to Jefferson Construction & Oil Treating Co. (U.S.P. 1,643,698—9, 27.9.27. Appl., 10.11.24. Renewed 1.3.27).—(a) Petroleum emulsion is treated with the sulphonic acid mixture obtained in the purification of oil (0·2%), and with an alkali or alkaline-earth chloride (1·5%) in solution. (b) The sulphonic acid mixture is replaced by a soap solution.
T. S. Wheeler.

Breaking of petroleum emulsions. M. De Groote and W. C. Adams, Assr. to W. S. Barnickel & Co. (U.S.P. 1,641,804, 6.9.27. Appl., 11.8.26).—The ammonium salt of the product obtained by heating a Twitchell reagent with oleic or hydroxystearic acid (1 pt.) is employed. T. S. Wheeler.

Treatment of organic liquids [hydrocarbons] with alternating electric discharge. Siemens & Halske A.-G. (F.P. 615,581, 4.5.26).—The hydrogen generated by the discharge through liquid hydrocarbons is used for boiling the liquid, thus causing frothing. L. A. Coles.

Briquetting of fuel. P. E. Welton and G. H. Wadsworth, Assrs. to P. E. Welton Engineering Co. (U.S.P. 1,642,055, 13.9.27. Appl., 25.11.22).—See E.P. 244,624; B., 1926, 181.

Treatment of oil. G. C. Kelley (Re-issue 16,778, 25.10.27, of U.S.P. 1,465,398, 21.8.23).—See B., 1923, 1012 a.

Manufacture of bituminous products etc. D. B. W. Alexander (E.P. 270,649, 7.3.27. U.S., 4.5.26).—See U.S.P. 1,603,502; B., 1927, 39.

Annular rotary-hearth ovens (E.P. 278,207).—See I.

Hydrogenation process (G.P. 441,164).—See XX.

III.—TAR AND TAR PRODUCTS.

Analysis of phenol (carbolic acid). S. del Mundo (Philippine J. Sci., 1927, **33**, 363—373).—A comprehensive and somewhat polemical discussion of the numerous modifications of Koppeschaar's original bromination method of analysing aqueous solutions of nearly pure phenol. Syrupy phosphoric acid is suggested as a better reagent than concentrated hydrochloric acid to liberate bromine from potassium bromide–bromate in countries such as the Philippines where normal laboratory temperature is 30°. The gravimetric method is inaccurate owing to uncertainty as to whether tribromophenol or tribromophenol bromide is precipitated.
E. Holmes.

Patent.

Extraction of neutral oils from tar, tar-oil, or pitch. H. Wittek (E.P. 256,923, 15.6.26. Ger., 12.8.25).—The acid constituents of crude coal tar are separated by dissolving the latter in an organic solvent (*e.g.*, alcohol, acetone) not soluble in paraffin oil, and agitating the solution with an oil composed mainly of hydrocarbons of the paraffin series. The neutral oils may be separated from the paraffin extract by subsequent distillation. The process may be carried out in the vapour phase or under pressure. R. C. Odams.

IV.—DYESTUFFS AND INTERMEDIATES.

Coal-tar colours in food. Nicholls.—See XIX.

Patents.

Manufacture of vat dyes of the anthraquinone series. I. G. Farbenind. A.-G., Assees. of F. Kačer (G.P. 439,614, 29.3.25. Addn. to G.P. 436,535; B., 1927, 387).—The processes of the prior patent are extended to anthraquinoneoxazoles. 2-Phenylanthraquinone-α-oxazole [6 : 7-phthaloyl-2-phenylbenzoxazole], obtained by heating 1-chloro-2-benzamidoanthraquinone with sodium carbonate in nitrobenzene, gives by nitration and reduction a red vat dye; the β-oxazole, from 2-bromo-1-benzamidoanthraquinone, yields a similar dye. Nitration and reduction also convert into vat dyes the oxazoles from 1 : 5-dichloro-2 : 6-dibenzamidoanthraquinone (brown), from 1-chloro-2-aminoanthraquinone (2 mols.) and teraphthalyl chloride (1 mol.; brown-red), and from 2-aldehydoanthraquinone and *o*-aminophenol (brown-red). Yellow vat dyes with good fastness properties are obtained by acylation (benzoylation) of the amino-oxazoles.
C. Hollins.

Manufacture of new vat dyes and intermediates for vat dyes. Brit. Dyestuffs Corp., Ltd., A. Shepherdson, and A. J. Hailwood (E.P. 278,102, 7.7.26).—Pyranthrone is oxidised with manganese dioxide in sulphuric acid. The product may be alkylated directly, or after reduction with sodium hydrogen sulphite, with or without removal of unchanged pyranthrone by extraction with nitrobenzene. Methylation gives a brown vat dye, fast to acids, alkalis, and chlorine.
C. Hollins.

Dyes and dyeing [soluble esters of leuco-vat dyes]. Morton Sundour Fabrics, Ltd., J. Morton, B. Wylam, J. E. G. Harris, and J. I. Morgan (E.P. 278,399, 3.5.26. Addn. to E.P. 251,491; B., 1926, 625).—The processes of the prior patent are performed in presence of a diluent, such as acetone or nitrobenzene.
C. Hollins.

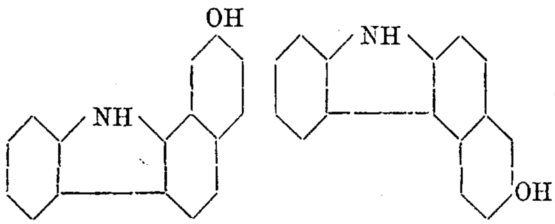
Manufacture of halogenation products of perylenetetracarboxylic di-imide and its derivatives. Kalle & Co. A.-G., Assees. of M. P. Schmidt and W. Neugebauer (G.P. 441,587, 27.10.22).—The di-imide and its derivatives (excluding those of G.P. 276,956) are halogenated in chlorosulphonic acid. The tetrachlorodi-imide, obtained by the action of chlorine in chlorosulphonic acid in presence of iodine, dyes cotton from the vat a yellow-red shade, becoming lilac on soaping.
C. Hollins.

Preparation of ice-colours. E. Gebauer-Fülnegg and W. Specht (Austr. P. 105,341, 14.11.24).—Diazo or tetrazo compounds are coupled with β-naphthols containing in the 1-position halogen, nitro-, alkyl, aryl, or acid groups. The substituents are not expelled, coupling occurring in the hydroxyl group. *p*-Nitrodiazobenzene is thus coupled with 1-chloro- or 1-bromo-β-naphthol (brown), 1-bromo-2-hydroxy-3-naphthoic acid (brown-orange), 1 : 3 : 6-tribromo-β-naphthol (orange), 1-nitro-β-naphthol (light brown), 1-methyl-β-naphthol (yellow-brown), 1-sulphomethyl-β-naphthol (yellow-red).
C. Hollins.

Manufacture of azo dyes. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning

(E.P. 255,900, 26.7.26. Ger., 25.7.25. Addn. to E.P. 231,529; B., 1925, 909).—4-Arylsulphonylalkyl- (or aralkyl-)amino-2-aminophenyl alkyl or aralkyl ethers, obtained by the action of an arylsulphonyl chloride on a 2-nitro-4-aminophenyl ether with subsequent alkylation or aralkylation and reduction, are diazotised and coupled in substance or on the fibre with a 2 : 3-hydroxynaphthoic arylamide. The dyes give redder shades than those of the prior patent. 4-Benzenesulphonylmethylamino-*o*-anisidine, diazotised and coupled with 2 : 3-hydroxynaphthoic 5-chloro-*o*-toluidide, gives a bordeaux-red. C. Hollins.

Manufacture of insoluble azo dyes. I. G. Farbenind. A.-G. (F.P. 617,211, 8.6.26. Ger., 26.6.25).—Hydroxynaphthacarbazoles are coupled in substance or on the fibre with unsulphonated diazo compounds. 2-Hydroxy-α-naphthacarbazole (I), obtained by alkaline

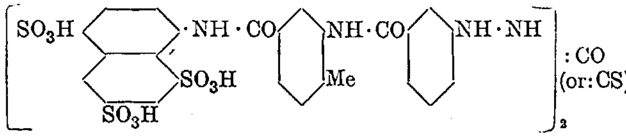


(I) (II)

fusion of the Bucherer reaction product from phenylhydrazine and α-naphthylamine-7-sulphonic acid, is coupled with diazotised *m*-nitroaniline (red-brown) or 4-nitro-*o*-anisidine (black-brown); 4-hydroxy-α-naphthacarbazole (II, from Brönner acid) with 4-nitro-*o*-anisidine (black-violet); 2-hydroxy-9-methyl-α-naphthacarbazole (as I) with 4-nitro-*o*-toluidine (bordeaux). C. Hollins.

Manufacture of disazo dyes. I. G. Farbenind. A.-G., Assees. of R. Kirchhoff, R. Haugwitz, and M. Cantor (G.P. 439,518, 16.5.25. Fr., 29.4.26).—Dyes suitable especially for weighted silk are obtained by condensing certain aminodisazo dyes with nitrobenzene derivatives containing a reactive substituent (*e.g.*, 1-chloro-2 : 4-dinitrobenzene, 1-chloro-4-nitrobenzene-2-sulphonic acid). The disazo dyes are those formed by coupling a 1 : 8-aminonaphtholsulphonic acid with 1 mol. of a suitable diazo compound and 1 mol. of a *m*- or *p*-nitrodiazo compound or of a nitrophenyl-*m*- or *p*-aminodiazo compound, the nitro-group being subsequently reduced. C. Hollins.

Manufacture of carbazides and thiocarbazides of the naphthalene series. Brit. Dyestuffs Corp., Ltd., G. M. Dyson, F. A. Mason, and A. Renshaw (E.P. 278,037, 25.5.26).—Carbazides or thiocarbazides obtained by the action of phosgene or of thiophosgene on hydrazines of the type, $P\cdot[NH\cdot X\cdot Q]_{n-2}\cdot NH\cdot Y\cdot R\cdot NH\cdot NH_2$, in which n is greater than 1, P is a sulphonated naphthalene or acenaphthene residue, Q and R are aryl, aralkyl, or aralkylene residues, and X and Y are carbonyl or sulphonyl groups, are found to be valuable agents for destruction of blood parasites whilst comparatively non-toxic to man. They are also intermediates for dyes. $P\cdot NH_2$ may be, *e.g.*, di- or tri-sulphonic acids of naphthylamines, aminonaphthols, 5-aminoacenaphthene, etc. $NH_2\cdot Q\cdot X\cdot OH$ and $NH_2\cdot R\cdot Y\cdot OH$ may be aminobenzoic, aminotoluic, aminocinnamic, aminophenylacetic, aminonaphthoic, anilinesulphonic, or naphthylaminesulphonic acids or their substitution products. The amine $P\cdot NH_2$ is first treated with a nitroaroyl chloride or a nitroarylsulphonyl chloride, and the nitro-group is reduced. A further nitroaroyl or nitroarylsulphonyl group may be introduced, followed by reduction to an amine, and this process repeated. Any of the amines so formed may be diazotised, reduced to a hydrazine, and finally treated with phosgene or thiophosgene. The preparation of *carbazido-* and *thiocarbazido-bis-2-(m-benzamido)-4-toluoyl-1′-naphthylamine-4′ : 6′ : 8′-trisulphonic acids* (annexed formula) is described.



C. Hollins.

Manufacture of dinaphthyldicarboxylic acids. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 278,100, 6.7.26).—Aminonaphthoic acids or esters are diazotised and decomposed by means of a suitable reducing agent, particularly ammoniacal cuprous oxide solution prepared by passing sulphur dioxide into a solution of copper sulphate and ammonia. 1 : 8-Aminonaphthoic acid is thus converted into 1 : 1′-*dinaphthyl*-8 : 8′-*dicarboxylic acid*, m.p. above 300° (*ethyl* ester, m.p. 183°). 2 : 2′-*Dinaphthyl*-3 : 3′-*dicarboxylic acid*, m.p. 290—292° (*ethyl* ester, m.p. 171°), 1 : 1′-dinaphthyl-2 : 2′-dicarboxylic acid, m.p. 266°, 1 : 1′-*dichloro*-2 : 2′-*dinaphthyl*-3 : 3′-*dicarboxylic acid*, m.p. 198°, 4 : 4′-*dibromo*-1 : 1′-*dinaphthyl*-8 : 8′-*dicarboxylic acid*, m.p. above 300°, the corresponding 4 : 4′-*dichloro*-compound, m.p. above 300° (*ethyl* ester, m.p. 194°), the 5 : 5′-*dichloro*-compound, the 5 : 5′-*dimethoxy*- and *diethoxy*-compounds, m.p. above 300°, and the 4 : 4′-*disulpho*-compound, are similarly prepared. 1-*Chloro-2-amino-3-naphthoic acid*, m.p. 254°, is obtained from 9-chloro-ββ-naphthisatin; 4-bromo-, 4-chloro-, and 5-chloro-1-amino-8-naphthoic acids from the corresponding naphthastyryls. C. Hollins.

Preparation of colouring matters [of the anthraquinone series]. L. J. Hooley, J. Thomas, and Scottish Dyes, Ltd. (E.P. 278,417, 2.7.26).—3-Chloro-1-bromo-2-aminoanthraquinone or 1 : 3-dichloro-2-aminoanthraquinone is condensed with primary amines (aniline, *p*-toluidine, methylamine) to give acetate silk dyes, which are converted by treatment with sulphuric acid or oleum into acid dyes for wool or silk. C. Hollins.

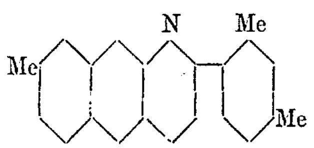
[Manufacture of] anthraquinone derivatives [for use as dyes and intermediates]. W. Smith, J. Thomas, and Scottish Dyes, Ltd. (E.P. 287,039, 26.5. and 10.6.26).—1-Halogeno-2-anthraquinonylurethanes are heated with a metal (copper) in presence or absence of a solvent (nitrobenzene) to give flavanthrones directly, or at lower temperatures to give 2 : 2′-diurethano-1 : 1′-dianthraquinonyls, which may be converted into flavanthrones by heating alone, or with concentrated sulphuric acid or alcoholic alkali, or in an inert solvent (nitrobenzene or naphthalene). 2 : 2′-*Diurethano*-1 : 1′-*dianthraquinonyl* melts above 250°. C. Hollins.

Manufacture of anthraquinone derivatives. I. G. FARBENIND. A.-G., Assees. of P. NAWIASKY (G.P. 440,891, 23.1.25).—Aromatic *p*-quinones and their derivatives, including halogen compounds, are condensed with monoacylated diaminoanthraquinones, or with diaminoanthraquinones and subsequently acylated. The product from 1 : 5-diaminoanthraquinone and α-naphthaquinone gives with benzoyl chloride an orange vat dye (yellow-brown on wool), with oxalyl chloride an orange, with adipyl chloride an orange (red-brown on wool), with nitroanthraquinone-2-carboxyl chloride a red vat dye; the 1 : 4-isomeride dyes bordeaux. Benzoylation of the product from 1 : 5-diaminoanthraquinone and *p*-benzoquinone gives a brown vat dye, becoming orange by treatment with hypochlorite. C. HOLLINS.

Manufacture of dyes and intermediates. R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 278,112, 13.7.26; cf. E.P. 251,313; B., 1926, 576).—The processes of the prior patent are improved by oxidising the benzanthrone at low temperatures. *E.g.*, a solution of manganese dioxide in 10 pts. of concentrated sulphuric acid at 100—110° is cooled to 0° and added to a solution of benzanthrone in sulphuric acid at 0°. C. HOLLINS.

Manufacture of dibenzanthronyls. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 278,047, 31.5.26. Addn. to E.P. 203,533; B., 1923, 1064 A).—The 4 : 4′-dibenzanthronyls of the prior patent are obtained by condensation of 4-halogenobenzanthrones with benzanthrones having free 4-position. The reaction proceeds n presence of alkaline condensing agents at 0—15°, or at 90—95°, and may be used for preparing unsymmetrical dibenzanthronyls. 4-Chlorobenzanthrone, m.p. 204°, and benzanthrone with sodioaniline in aniline give 4 : 4′-dibenzanthronyl. 9-*Methyl*- (m.p. 330—331°), 9-*chloro*- (m.p. 312—314°), and 10-*chloro*-, (m.p. 305°) -*dibenzanthronyls* are obtained similarly from 4-chlorobenzanthrone and 9-methyl-, 9-chloro-, and 10-chlorobenzanthrones, respectively. 4 : 9- and 4 : 10-Dichlorobenzanthrones (the former prepared from 2 : 6-, the latter, m.p. 278°, from 2 : 7-dichloroanthraquinone) condense with 6- and 7-chlorobenzanthrones to give, respectively, 9 : 9′- and 10 : 10′-*dichlorodibenzanthronyls*. C. HOLLINS.

Manufacture of heterocyclic compounds. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 258,553, 14.7.26. Ger., 15.9.26. Addn. to E.P. 251,270; B., 1927, 742).—The process of the prior patent is extended to the manufacture of polynuclear heterocyclic systems. A heterocyclic ketone, carbinol, or methane, in which there is an *ortho*-methyl or -methylene group, is cyclised by passing its vapour, in presence or absence of an oxidant, over a hot porous catalyst (active carbon). The hydroxymethylene derivative of *m*-4-xylyl methyl ketone, when heated with ammonium acetate (cf. G.P. 418,218; B., 1926, 216), gives 2-(4′-m-*xylyl*) - 5 - *pyridyl* 4-m-*xylyl ketone* (*sulphate*, m.p. 220—221°), which is cyclised by being passed with air over active carbon at 400—420° to form 2-(4′-m-*xylyl*)-8-*methyl*-1-*anthrapyridine* [2-(4′-m-*xylyl*)-8-*methyl*-ββ-*naphthaquinoline*], m.p. 172°. C. HOLLINS.



Manufacture of chlorides of *o*-hydroxycarboxylic acids of polynuclear hydrocarbons. BRIT. SYNTHETICS, LTD., and E. B. HIGGINS (E.P. 278,463, 20.7.26).—The dry monosodium salt of a polynuclear aromatic *o*-hydroxycarboxylic acid (2 : 3-hydroxynaphthoic acid) is ground with one equivalent of thionyl chloride below 60°, *e.g.*, in a ball-mill for 10 min. On removal of sulphur dioxide in a current of air, 2-hydroxy-3-naphthoyl chloride is obtained pure and stable in quantitative yield. C. HOLLINS.

Catalytic oxidation of organic compounds. I. G. FARBENIND. A.-G. (G.P. 441,163, 15.12.22).—A succession of catalysts is employed. The first catalyst should be active at moderate temperatures, and the heat of reaction heats the gases to a temperature suitable for the next catalyst. The rate of oxidation is thus moderated by the use of less active catalysts as the temperature rises. *E.g.*, naphthalene vapour and air are led first over vanadic acid on metal granules at 320°, then over vanadic acid on powdered kieselguhr at 350—410°, phthalic anhydride being obtained in good yield. In some cases it is desirable to conduct the oxidation in stages in order to prevent overheating; *e.g.*, acenaphthene is oxidised first to acenaphthylene, and then to naphthalic acid. C. HOLLINS.

Reduction of organic compounds. I. G. FARBENIND. A.-G., Assees. of M. MÜLLER-CUNRADI, M. LUTHER, and K. PIEROH (G.P. 441,179, 18.1.25).—The reducing agent may be prepared by heating iron pentacarbonyl with methyl-alcoholic potassium hydroxide under reflux. On adding nitrobenzene and distilling off the methyl alcohol, aniline is obtained by steam distillation. If iron pentacarbonyl is added slowly to a warmed mixture of nitrobenzene and dilute potassium hydroxide solution, carbanilide and aniline are obtained. Vat dyes of the indigo, thioindigo, anthraquinone, and quinone groups, sulphide dyes, and azines are reduced to leuco-compounds by adding iron pentacarbonyl to a stirred aqueous alkaline suspension of the vat dye etc. at 60—70°. Azoxybenzene and hydrazobenzene may be obtained from nitrobenzene, *o*-aminophenol quantitatively from *o*-nitrophenol, and benzoin from benzil. C. HOLLINS.

Manufacture of aryl esters of nitroaminobenzenesulphonic acids. W. DUISBERG, W. HENTRICH, and L. ZEH, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,646,785, 25.10.27. Appl., 9.6.25. Ger., 14.6.24).—See E.P. 235,598; B., 1925, 704.

Manufacture of dinitroperylenequinone. A. ZINKE, Assr. to F. BENSA (U.S.P. 1,642,263, 13.9.27. Appl., 15.6.23. Austr., 20.6.22).—See E.P. 199,720; B., 1923, 1170 A.

Decolorisation of aqueous liquids (E.P. 278,485). **Friction surfaces** (E.P. 278,465).—See I.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Action of atmospheric influences on fibrous material. H. SOMMER (Chem.-Ztg., 1927, 51, 777—778).—Samples of different fabrics (wool, cotton, silk, hemp, etc.) were exposed to the weather in both clean and

polluted atmospheres for periods of 3, 6, 9, and 12 months, the total number of hours' sunshine being recorded for each period of exposure. With woollen fabrics, dyed and undyed, atrophy of the hairs on the side exposed to the light was observed after three months, the other side being unaffected. Deterioration of the wool was indicated by loss in weight and strength of the material, and was approximately proportional to the number of hours of sunshine; it is attributed in all cases to the action of rays of short wave-length. The presence of sulphuric acid due to the oxidation of contained sulphur was shown. Moisture also accelerates the process and increases the loss in weight by causing the decomposition products to go into solution. The extent of the damage depends also on the thickness and porosity of the material. Pre-treatment of the wool with sulphuric acid retards the destruction, whilst treatment with alkalis hastens it. Dyeing, according to the dyestuff used, exerts a more or less protective action which is approximately proportional to the depth of the dye. Chroming and treatment with copper sulphate result in increased protection. The strength of raw, degummed, and weighted silk was reduced to one tenth of its original value by exposure for three months, a marked loss in weight being also observed; fabrics from vegetable fibres behaved similarly. Besides the destruction of the cuticle of cotton and of the incrusting substances of bast fibres, the cellulose itself undergoes a change which is indicated by the solubility of the modified material in boiling sodium hydroxide, a behaviour analogous to that of oxycellulose. The change is ascribed not to an oxidation process, but to the formation of "photocellulose." If the sensitiveness to weathering be judged by the number of hours of sunshine necessary to reduce the original strength of the fabric to half its value, the materials (not allowing for their unequal thicknesses) may be arranged in the following decreasing order: silk, jute, artificial silk, cotton, flax, hemp, raw wool, and chromed wool.

B. P. RIDGE.

Influence of the preliminary alkaline treatment and of the time of chlorination in obtaining cellulose by the chlorine process. I. MUTTI and M. VENTURI (Annali Chim. Appl., 1927, **17**, 391—401).—When, in the manufacture of cellulose by the chlorine process, a high yield is desired, 1% sodium hydroxide solution should be used in the preliminary treatment of the material, whereas when a purer product in lower yield is required, 4% sodium hydroxide solution should be employed. In the former case the action of the chlorine should not be too prolonged, in order that the resulting product may not suffer injury; in the latter case, however, no such injury results, and the products obtained contain up to 86% of α-cellulose and are well suited to the manufacture of artificial silk. Increase of the concentration of the sodium hydroxide solution used in the preliminary treatment from 5 to 10% diminishes the final yield without improving the quality of the cellulose obtained.

T. H. POPE.

Reactions relating to carbohydrates and polysaccharides. XII. Action of chromic acid on cotton cellulose. H. HIBBERT and S. M. HASSAN (J.S.C.I., 1927, **46**, 407—411 T; cf. Hibbert and Parsons, *ibid.*, 1925, **44**, 473 T).—Dextrose and α-methylglucoside on oxidation with chromic acid in presence of 3—6% sulphuric acid, using a ratio of 2 atoms of "active oxygen" per dextrose mol., yield formic acid and a furfuraldehyde-yielding constituent in the ratio 2 : 1, but the amount of glycuronic acid formed in both cases is small, and α-methylglucoside probably undergoes hydrolysis under these conditions. Acetic acid was not detected among the oxidation products. Lactose with chromic acid over a wide range (up to about 8 atoms of "active oxygen" per mol.) gives a constant amount of furfuraldehyde-forming constituent, indicating the presence of an intermediate which is stable towards the oxidising agent. Cotton cellulose when treated with chromic acid (2 atoms of "active oxygen" per C_6 complex) in presence of sulphuric acid of varying concentration (10—50%) gives a more degraded product in decreasing yield with increase in acid concentration, but the copper number of the product remains practically constant whilst the furfuraldehyde value decreases, showing that such values are not suitable for the determination of the character of an oxidised cellulose. Cotton cellulose on oxidation with varying amounts of chromic acid (0·25—3 atoms of "active oxygen") in 10% sulphuric acid solution gives a high yield of an oxidised cellulose (94·6—82%), and the product has approximately the same copper number and furfuraldehyde and Lefèvre carbon dioxide values, indicating a gradual progressive oxidation of the fibre surface. Gluconic and saccharic acids do not evolve carbon dioxide when boiled with 12% hydrochloric acid nor reduce Fehling solution.

W. J. POWELL.

Use of waste wood products. SCHWALBE.—See II.

Sulphite-cellulose waste liquor as manure. GÖRBING.—See XVI.

PATENTS.

Manufacture of artificial silk [from cotton rags]. J. UMBACH (E.P. 278,131, 5.8.26).—Viscose silk having 30% higher elasticity than silk prepared from fir wood sulphite-pulp or cotton linters is manufactured from waste coloured cotton rags, previously purified by digestion with caustic alkalis under pressure at a high temperature. A satisfactory treatment consists in heating the rags for 6—8 hrs. under 4—5 atm. pressure (240—250°) in a 1% solution of caustic soda; subsequent bleaching is unnecessary.

A. J. HALL.

Conditioning [oiling of] textile fibres. R. B. SMITH, Assr. to H. L. SIEVER (U.S.P. 1,642,092, 13.9.27. Appl., 28.6.26; cf. U.S.P. 1,550,396; B., 1925, 842).—Raw cotton or other textile stock is treated with oil atomised with steam.

T. S. WHEELER.

Manufacture of a composition for treating [fireproofing] fibre products. C. R. FELIX (U.S.P. 1,643,116, 20.9.27. Appl., 6.3.25).—Cotton, paper, etc. is immersed at 85° in a solution containing sodium silicate, sodium tungstate, and sodium carbonate or boric acid, according as a non-flexible or flexible product is required.

T. S. WHEELER.

Treatment [delustring] of cellulose fibres or filaments. B. BORZYKOWSKI (E.P. 261,333, 27.4.26. Ger., 11.11.25).—Viscose silk having a natural silk-like sheen is obtained by bleaching crude viscose before

desulphurisation, and afterwards treating it with a 1—2% solution of an aluminium, magnesium, or zinc salt.
A. J. HALL.

Application of cellulose ethers or esters [dioxan as a solvent]. I. G. FARBENIND. A.-G. (E.P. 275,653, 15.12.25. Ger., 23.12.24).—1 : 4-Dioxan (diethylene dioxide) dissolves cellulose ethers or esters with or without addition of resins, dyes, and/or other solvents; the solutions are useful for spinning artificial silk or as varnishes. C. HOLLINS.

Treatment of nitrocellulose [for lacquers] to render it safe during storage or transport. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 252,382, 20.5.26. Ger., 20.5.25). —Lacquer nitrocellulose (of a comparatively low degree of nitration) is moistened with butyl alcohol or a higher alcohol, *e.g.*, amyl alcohol or glycerol, to render it safe during transport or storage and facilitate subsequent treatment in the manufacture of lacquers.
S. S. WOOLF.

Process for bleaching [wood pulp]. C. B. THORNE (U.S.P. 1,643,566, 27.9.27. Appl., 16.10.25).—Wood pulp, concentrated to 15—20%, is introduced at a constant rate at the top of a tower, in which it is treated with bleaching material, being meanwhile agitated mechanically and aerated. The bleached material is withdrawn at the bottom of the tower and washed.
T. S. WHEELER.

Batting process [for fibres]. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,646,605, 25.10.27. Appl., 18.11.22).—See E.P. 232,763; B., 1925, 515.

Manufacture of artificial silk. M. HÖLKEN, JUN. (U.S.P. 1,641,588, 6.9.27. Appl., 25.1.23. Ger., 12.7.20).—See E.P. 211,691; B., 1924, 374.

Manufacture of paper pulp. B. S. SUMMERS (U.S.P. 1,643,826, 27.9.27. Appl., 22.12.23).—See Can. P. 246,537; B., 1926, 153.

Separation of the fibre from the pulp of sisal or like leaves. HYDRAULIC ENGINEERING CO., LTD., and E. D. RUTHERFOORD (E.P. 278,457, 14.7.26).

Manufacture of twisted artificial silk from cuprammonium cellulose solutions by the stretch-spinning process. J. P. BEMBERG A.-G. (E.P. 260,564 and Addn. E.P. 268,393, [A] 1.10.26, [B] 28.3.27. Ger., [A] 2.11.25, [B] 27.3.26).

Manufacture of finely-perforated ceramic plates for use in the manufacture of artificial silk etc. J. F. SCHEID, V. TOUNDORF, and C. ZEISS (E.P. 278,097, 3.7.26).

Yeast (F.P. 614,037).—See XVIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Process for "brightening" [dyed] silk. K. WOLFGANG (Kunstseide, 1927, 9, 517—518).—Much more permanent results are obtained in the usual process for brightening dyed silk by treatment with a soap and an organic acid if oleic acid is added to the potash-olive oil soap usually employed. This improved process has no deleterious effect on the elasticity and durability of the silk. A. J. HALL.

Light sources for testing the fastness of dyes. I. H. HADFIELD (J. Text. Inst., 1927, 18, 527—558 T).—A summary comprising information published prior to December, 1925, bearing on the suitability of various artificial sources as standard lamps for dye-fading tests.
B. P. RIDGE.

PATENTS.

Dyeing of vegetable fibre. N. N. VOROSHTZOV, and STAATLICHE VEREINIGTE ANILINFABR. (G.P. 441,053, 24.11.22. Russia, 23.3.21).—Cotton is dyed from an alkaline bath with mono-, dis-, or poly-azo dyes containing 6-nitro-β-naphthylamine-8-sulphonic acid as first component. With α-naphthol-4-sulphonic acid this gives a violet-red; with α-naphthylamine-6(or 7)-sulphonic acid a grey-violet, which may be diazotised on the fibre and developed to blue to black shades.
C. HOLLINS.

Improvement of washing- and milling-fastness of acid dyeings on wool. I. G. FARBENIND. A.-G., Assees. of F. JUST (G.P. 440,606, 31.1.24).—Acid wool dyeings are after-treated with nitrous acid.
C. HOLLINS.

Fixation of basic dyes on cotton. I. G. FARBENIND. A.-G., Assees of A. GÜNTHER, J. HALLER, and E. KÖSTER (G.P. 441,326, 20.2.25).—Arylamides of aromatic hydroxy-acids (*e.g.*, 2 : 3-hydroxynaphthoic β-naphthylamide, 1 : 2-*hydroxynaphthoic β-naphthylamide*, m.p. 180°, disalicylbenzidine) are used as mordants for basic colours, and give fast dyeings without after-treatment with tartar emetic. The colour bath is exhausted better than with tannin, and deeper shades are obtainable.
C. HOLLINS.

[Dyeing of "mixed" cellulose acetate] threads, cords, and fabrics. BRIT. CELANESE, LTD., C. W. PALMER, and S. M. FULTON (E.P. 278,116, 21.7.26).—Yarns and fabrics manufactured so that they contain threads of normal and delustred cellulose acetate silk have special advantages in handle and appearance. Since the process of delustring (*e.g.*, treatment of the silk with wet steam at 100°) decreases the affinity of cellulose acetate silk for basic dyes such as Capri Blue, two-colour effects may be obtained by suitably dyeing the manufactured products. A. J. HALL.

Dyeing of cellulose esters. I. G. FARBENIND. A.-G., Assees. of H. EICHWEDE, E. FISCHER, and C. E. MÜLLER (G.P. 441,325, 28.9.24).—Cellulose esters (*e.g.*, acetate silk) are dyed with monoazo dyes from diphenylamine-monosulphonic acid coupled with unsulphonated diazo compounds of the benzene or naphthalene series. *E.g.*, *m*-nitroaniline → diphenylaminesulphonic acid gives a golden yellow. C. HOLLINS.

Printing and fixing basic dyes on acetate silk. I. G. FARBENIND. A.-G., Assees. of A. SCHNEEVOIGT (G.P. 441,327, 18.4.25).—Clear prints are obtained by the use of phenols (phenol, naphthols, resorcinols, cresolsulphonic acids) in printing or fixing pastes with basis colours. The addition of tannin and after-treatment with tartar emetic are unnecessary. C. HOLLINS.

Vat dyes and dyeing (E.P. 278,399).—See IV.

Bleaching of wood pulp (U.S.P. 1,643,566).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Blattner process for the preparation of caustic soda from sodium carbonate. HIRCHBERG (Chem.-Ztg., 1927, **51**, 765).—Caustic soda can be produced in nearly theoretical yield by heating a mixture of 150 pts. of iron oxide with 100 pts. of sodium carbonate, such process being a modification of that described in E.P. 203,271 (B., 1923, 1070 A). The reaction is hastened by the use of a catalyst. Sodium carbonate of varying grades of purity can be used, from the pure material obtained from the ammonia–soda process to a naturally occurring carbonate containing 12% of sodium chloride and 10% of sodium sulphate. The carbon dioxide evolved during the process is treated with sodium carbonate in absorption chambers or brought into contact in towers with a solution of the same. Sodium bicarbonate is produced from which pure carbon dioxide and sodium carbonate can be regenerated. The use of a rotary furnace filled completely with the reaction material is not considered suitable. An economical type of oven is suggested in which the heating is carried on continuously, the finely-ground and well-mixed material being introduced in a thin layer at one end and carried directly into water at the other. R. C. ODAMS.

[Catalytic] oxidation of ammonia. F. RASCHIG (Z. angew. Chem., 1927, **40**, 1183—1185).—Andrussov and Bodenstein's view (B., 1927, 217) that nitroxyl, NHO, is the primary product of the oxidation of ammonia and that it decomposes into nitric oxide and hydrogen is criticised as being without experimental foundation and contrary to the known properties of this compound. The author's view that imide, NH, is the intermediate product is reiterated, and the details of the experiment in which he found hydrazine in the gases from the combustion of ammonia with insufficient oxygen ($NH_3 + NH = N_2H_4$) are described. The combustion was effected by a coal-gas flame within a flask fed with ammonia and oxygen in regulated amounts and containing ammonia solution. The hydrazine was determined by titration with iodine or as benzylideneazine. C. IRWIN.

The ferric sulphate–sulphuric acid process. O. C. RALSTON (U.S. Bur. Mines Bull. 260, 1927, 1—61). —Ferric salts are suitable for leaching low-grade ores of copper and other metals, dissolving sulphides as well as oxidised ores. Hitherto, however, no economical method has been known of oxidising the resulting ferrous salts for further use. It has been discovered that ferrous iron and sulphur dioxide catalyse each other's oxidation. When sulphur dioxide diluted with air is passed through a solution of ferrous sulphate the reaction $2FeSO_4+SO_2+O_2=Fe_2(SO_4)_3$ occurs. When oxidation of ferrous sulphate has reached its limit for the given conditions, oxidation of sulphur dioxide to sulphuric acid still proceeds up to a certain concentration. These two reactions were studied in detail. The most satisfactory apparatus was found to be a tall glass jar filled with solution with means for passing in bubbles of gas of size not above 1 mm. at the bottom. On a larger scale a high wooden tank was used. It was found that if the ferrous sulphate solution became acid, which only happened in inefficient apparatus, the rate of oxidation of iron fell off. If the solution was initially acid, a lag in the start of the reaction occurred. After all the iron has been oxidised formation of acid can proceed, at economic rate, up to a concentration of 110 g. of sulphuric acid per litre provided the solution contains 0·5—1·0 g. of ferrous iron per litre. Any concentration of ferrous iron up to saturation can be oxidised. For the oxidation to be complete, however, an optimum ratio of sulphur dioxide to oxygen is necessary. The maximum percentage of sulphur dioxide for 1 mm. bubbles is 7% by volume. For sulphuric acid synthesis after the oxidation of the iron is complete this can be increased. The velocity of the reaction is inversely proportional to the square root of the diameter of the orifice producing the bubbles. With increase of temperature it tends to reach a maximum at about 40°. Increase of pressure gives only a small effect. Copper if present is detrimental to the reaction, and in any application to leaching liquors for copper ore will require removal. If gases from burning sulphur are to be used the desirable concentration will be 5·5% SO_2, from pyrites 5·0% SO_2 (in each case for iron oxidation). For a 10 ft. column with a diaphragm containing perforations of 0·118 mm. diam. (required to give 1 mm. bubbles) at a space velocity of 500 c.c. of gas per litre per min. the total pressure required is calculated to be 4·93 lb./in.^{2} The total power requirements of the process for blowing are estimated at 3·0—3·5 kw. per 1000 lb. of copper treated per 24 hrs. C. IRWIN.

Production of small bubbles of gas in liquids. MAIER.—See I.

Wood preservatives. CURTIN.—See IX.

PATENTS.

Treatment of crude nitrate of soda (caliche). I. G. FARBENIND. A.-G. (E.P. 266,735, 24.2.27. Ger., 24.2.26).—Caliche is subjected to prolonged treatment at ordinary temperature with a quantity of water such that the sodium nitrate is almost completely extracted but a large portion of the other soluble salts remains undissolved; the resulting solution is then used to leach at 100° a further quantity of caliche, after which it is cooled so that the sodium nitrate separates out. Instead of using fresh caliche for cold-leaching purposes, residues from any warm-leaching process may be employed, and instead of water for the cold-leaching, the solution remaining after the deposition of nitrate crystals may be used. W. G. CAREY.

Separation of native sodium salts, *e.g.*, thénardite and glauberite, from the accompanying rock salt gangue. F. STEIN (G.P. 442,646, 21.10.24).—The crude salt mixture is leached with water or liquors from previous operations in such a way that the rock salt dissolves leaving a residue of sodium sulphate or glauberite. If water is used the crude salt mixture is treated continuously on the counter-current principle with a rapid stream of very cold water. If the mixture contains an excess of rock salt the mother-liquors from the manufacture of Glauber's salt may be used, in which case most of the sodium sulphate in the liquors is deposited together with the insoluble sodium sulphate in the crude salt; the sodium chloride solution so obtained

may be freed from the calcium and sodium sulphate it contains by seeding with artificial glauberite. If the mixture contains an excess of sodium sulphate an aqueous suspension of gypsum dissolves the sodium chloride, whilst the sodium sulphate is converted into glauberite; alternatively, gypsum may be added together with mother-liquors from Glauber's salt.

A. R. Powell.

Manufacture [purification] of sodium phosphate. H. Howard, Assr. to Grasselli Chemical Co. (U.S.P. 1,642,244, 13.9.27. Appl., 31.10.22).—Crude sodium phosphate solution obtained in the manufacture of the salt from phosphate rock is decolorised by treatment with chlorine. T. S. Wheeler.

Production of sodamide. J. F. Wait, Assr. to Nat. Aniline & Chemical Co., Inc. (U.S.P. 1,646,372, 18.10.27. Appl., 18.12.24).—Gaseous ammonia is cooled below 5° and passed through molten sodium.

H. Royal-Dawson.

Manufacture of ammonium fluorides. J. W. Proctor, Assr. to Gen. Chemical Co. (U.S.P. 1,642,788, 20.9.27. Appl., 5.1.23).—Saturated ammonium fluoride solution is treated in a cycle first with hydrogen fluoride and then with ammonia. T. S. Wheeler.

Precipitation of heavy metals from ammoniacal solutions. I. G. Farbenind. A.-G. (E.P. 269,164, 31.3.27. Ger., 9.4.26).—The solutions are treated with carbon monoxide, hydrogen, or mixtures containing these gases under pressure and at an elevated temperature. By the application of different temperatures and pressures, several metals may be precipitated separately in succession. Noble metals may be precipitated either under increased pressure and at normal temperatures, or at an elevated temperature and under normal pressure.

M. E. Nottage.

Preparation of cyanogen compounds [calcium cyanide]. R. W. Poindexter, jun. and W. E. Olberg, Assrs. to California Cyanide Co., Inc. (U.S.P. 1,642,694, 20.9.27. Appl., 19.8.26; cf. U.S.P. 1,573,732; B., 1926, 486).—Anhydrous hydrocyanic acid is treated with calcium carbide in presence of a suitable catalyst, *e.g.*, ammonium bromide, thiocyanate, or iodide, or calcium bromide. T. S. Wheeler.

Treatment of lead chloride or basic chloride and application thereof to the winning of lead from ores, residues, etc. S. C. Smith, and Chemical & Metallurgical Corp., Ltd. (E.P. 278,093, 2.7.26).—The process of E.P. 237,308 (B., 1925, 758) is modified in that carbon dioxide, which is passed into a suspension of lead chloride in ammonia, is added at such a rate that it forms lead carbonate as soon as it enters the suspension so that dissolved carbon dioxide is avoided.

W. G. Carey.

Preparation of aluminium sulphate, free from iron, from alum. Vereinigte Aluminium Werke A.-G. (G.P. 442,596, 13.9.23).—The ammonium or potassium alum obtained by treating clay or other aluminous material with ammonium or potassium sulphate is decomposed with 50—60% sulphuric acid solution and the precipitated aluminium sulphate, after washing with more sulphuric acid, is freed from excess acid by agitation with a cold saturated solution of aluminium sulphate obtained in a previous operation. A. R. Powell.

Production of silicates. G. Herfeldt (G.P. 442,717, 29.4.24).—Mixtures of silicates with oxides such as lime and magnesia interact with water under the influence of the heat of reaction and in the presence of colloids capable of accelerating the reaction. L. A. Coles.

Production of normal and other standard solutions. E. de Haen Chem. Fabr. "List" G.m.b.H. (G.P. 442,706, 23.6.20).—Suitable weights of the desired materials are stored in air-tight bottles, and the contents are dissolved and made up to the correct volume when the solutions are required for use. L. A. Coles.

Granules for the absorption of [acidic] gases. L. A. Levy (E.P. 277,540, 25.1.27).—Hydrated barium hydroxide (270 pts.) is mixed with slaked lime (180 pts.), caustic soda or potash (15 pts.), and, if desired, activated charcoal (49 pts.; 100-mesh). Preferably the soda is dissolved in water (25 pts.) and the barium hydroxide added, the mass being then heated until fluid, whereupon the lime and charcoal, intimately mixed, are stirred in and the mass on cooling is granulated. H. Holmes.

Catalytic oxidation of carbon monoxide. I. G. Farbenind. A.-G. (E.P. 265,624, 7.2.27. Ger., 8.2.26).—Active adsorbent substances of gel character, *e.g.*, silica, stannic acid, etc., are used to eliminate vapours and gases injurious to catalysts used in the oxidation of carbon monoxide to the dioxide. H. Royal-Dawson.

Continuous distillation of crude carbon disulphide to obtain pure carbon disulphide. I. G. Farbenind. A.-G., Assees. of Chem. Fabr. Griesheim-Elektron (E.P. 260,236, 8.10.26. Ger., 24.10.25).—The rectification is carried out in two similar columns in series fitted with stills and reflux condensers. The first column is irrigated with crude carbon disulphide and the second with partially purified carbon disulphide from the first column, while the still of the first column is kept at a temperature above the m.p. of sulphur and that of the second slightly above the b.p. of pure carbon disulphide. The carbon disulphide vapours are discharged from the lower ends of the columns and are led to the level of the upper ends prior to condensation, while the waste gases (*e.g.*, hydrogen sulphide) issuing from the reflux condensers are further cooled and the carbon disulphide obtained thereby is again used for irrigating the first column. The sulphur separating from the carbon disulphide is withdrawn as a liquid from the still of the first column. W. G. Carey.

Treatment of sulphur. J. W. Schwab, Assr. to Texas Gulf Sulphur Co. (U.S.P. 1,646,838, 25.10.27. Appl., 30.1.24).—The normal colour of discoloured sulphur is restored by passing the molten material through a layer of fuller's earth. H. Royal-Dawson.

Oxidation of ammonia by means of oxygen. F. G. Liljenroth (E.P. 276,295, 26.11.26. Swed., 19.8.26).—See F.P. 610,010 and 610,160; B., 1927, 251.

VIII.—GLASS; CERAMICS.

Determination of sulphur present as sulphide in glass. H. Heinrichs (Z. anorg. Chem., 1927, **166**, 299—305).—Sulphides in glass may be determined by warming 1 g. of the finely-powdered glass with a mixture of 5 c.c. of water, 25 c.c. of dilute (1 : 1) hydrochloric acid, and 5 c.c. of 48% hydrofluoric acid in an apparatus

of sulphide-free glass, and passing the hydrogen sulphide evolved into $0 \cdot 01N$-iodine solution. Before the acid is added, the air is displaced from the apparatus with a stream of carbon dioxide, which is continued until all the hydrogen sulphide has been expelled. As, in general, the amount of sulphide present is very small, it is desirable to make a separate qualitative test in which the hydrogen sulphide is detected with a lead acetate paper. Analyses made in this way with various glasses show that the effect of a sulphide on the colour of glass is very much greater than that of an equal amount of the corresponding oxide. R. CUTHILL.

Refractories for the electric steel furnace. E. KOTHNY (Feuerfest, 1927, **3**, 157—164).—The characteristics of fourteen different refractories are described and their actual or possible application in electric furnace construction is indicated. The relative costs per cub. m. of brickwork are tabulated for six different types of refractory bricks. Silica bricks may be used in all parts of radiation furnaces and above the slag line in basic furnaces. By reason of their comparatively low price, they constitute one of the chief materials for electric furnace construction. Magnesite bricks are sensitive to abrupt changes of temperature; they are only applied where their slag-resisting property can be utilised. Calcined magnesite and calcined dolomite are suitable for basic furnaces. Chromite bricks may be used to form a neutral layer between acid and basic brickwork. Carborundum bricks are destroyed by the action of slags, and can therefore only be used in the roofs of furnaces. Corundum bricks are very refractory and resistant to temperature changes. Magnesia spinel bricks are very refractory and highly resistant to basic slags and to changes of temperature. Sillimanite bricks are attacked by acid slags. Fused magnesite is suitable for low-frequency induction furnaces. Zirconia bricks have a very high m.p. (3000°), high refractoriness under load (2500°), and are very resistant to basic slag and to temperature changes. The disadvantage of the last-mentioned seven materials (chromite—zirconia) is their high cost as compared with silica. Zirconia, however, may possibly find permanent application in the construction of high-frequency induction furnaces. F. SALT.

PATENTS.

[Flame-]tinting of glass. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of D. A. DEWEY and C. D. SPENCER (E.P. 271,481, 18.5.27. U.S., 18.5.26).—Glass which, when used for incandescence lamps etc., reproduces the effect of an oil or gas flame is prepared from an iron-manganese amber batch. Thus, *e.g.*, to each 100 lb. of a standard soda-lead batch are added 5 lb. of nitre, 3 lb. of ferric oxide, and 4 lb. of manganese dioxide. A. COUSEN.

Manufacture of stoneware highly resistant to perforation by the electric spark. F. SINGER (U.S.P. 1,642,754, 20.9.27. Appl., 18.12.26. Ger., 13.11.25).—Titanium compounds, *e.g.*, titanium dioxide, are added to the ingredients usually employed in the manufacture of ceramic insulating materials, and the mixture is fired, first in an oxidising and then in a reducing atmosphere. T. S. WHEELER.

Unburned refractory [magnesite] brick. G. K. SCHLOTTERER and R. H. YOUNGMAN, Assrs. to HARBISON-WALKER REFRACTORIES CO. (U.S.P. 1,643,181, 20.9.27. Appl., 15.5.26; cf. U.S.P. 1,564,394; B., 1926, 192).—A mixture of dead-burned magnesite and powdered sodium silicate (5—10%) is treated with water, formed, and slowly dried at 55—95° in an atmosphere of progressively decreased humidity. T. S. WHEELER.

Manufacture of glass. B. LONG, Assr. to SOC. ANON. DES MANUF. DES GLACES & PROD. CHIM. DE SAINT-GOBAIN, CHAUNY & CIREY (U.S.P. 1,633,534, 21.6.27. Appl., 12.1.26).—See E.P. 264,495; B., 1927, 523.

IX.—BUILDING MATERIALS.

Sintered alumina cements from Hungarian bauxite. J. VARGA (Z. angew. Chem., 1927, **40**, 1164—1167).—The recently discovered Hungarian bauxite deposits are in two fields, that of Gánt containing 10—15% Fe_2O_3 and 5—12% ($SiO_2 + TiO_2$) and that of Halimba containing 23—31% Fe_2O_3 with 1·4—4·1% ($SiO_2 + TiO_2$). Both types on fusion with limestone gave a good sintered cement with m.p. as low as 1250°. A large graphite crucible heated by a blowpipe was used. The time of fusion was without influence on the result. These cements set in a few hours and suffered no loss in strength in a 12 months' test. The Halimba bauxite gave best results when the fusion was in a reducing atmosphere. If the temperature is raised a partial reduction of oxide to metallic iron takes place. The cement then produced has a higher m.p., greater tensile strength, and is slower setting. Less heat is required completely to fuse these aluminous cements than to sinter ordinary Portland cement. C. IRWIN.

Influence of aluminium and zinc on cement. C. R. PLATZMANN (Chem.-Ztg., 1927, **51**, 833—835).—Porous concrete was made by mixing cement and water with aluminium or zinc powder (U.S.P. 1,087,098) or with zinc powder and calcium chloride (G.P. 327,907; B., 1921, 180 A). The metal formed an aluminate or zincate of calcium with evolution of hydrogen which rendered the concrete porous. Mixtures of 100 pts. of Portland cement, 50 pts. of water, and 0·1—0·25 pt. of aluminium powder showed volume increases for different cements varying from 68 to 127%. The use of more than 0·25% of aluminium powder did not result in a larger increase of volume, as the excess of aluminium remained uncombined. The use of cement-sand mixtures gave smaller volume increases, but an increase of 100% was obtained with a mixture of cement with 75% of coke ash. The density of the concrete increased with the amount of sand used, and addition of more than 40% of sand resulted in a rapid diminution in the crushing strength. The use of zinc powder alone gave volume increases of only 30—50%. Addition of 2¼% of zinc powder and 3¾% of calcium chloride to the cement and water yielded a concrete of high porosity and crushing strength. F. R. ENNOS.

Use of fluorspar in the cement industry. H. BECKER (Zement, 1927, **16**, 305—308; Chem. Zentr., 1927, I, 3219).—The liquid phase of clinker ceases at 1270—1300°, but with the addition of calcium fluoride the solidifying point becomes 900°. Rapid cooling is

necessary to prevent running of the clinker; sintering, therefore, is facilitated by the cautious addition of calcium fluoride. W. G. Carey.

Wood preservation. II. Arsenites of copper and zinc. III. Preservative properties of basic substances. L. P. Curtin (Ind. Eng. Chem. 1927, **19**, 993—999, 1159—1161; cf. B., 1927, 750).—II. Owing to the production of acid by wood-rotting fungi, salts as wood preservatives were tried which are of low solubility and which are not leached out by rain, but which are soluble in the acid solutions of p_H 5 or less produced by the fungi. Basic cuprous chloride made by reducing copper sulphate with sodium bisulphite in presence of a large amount of sodium chloride is precipitated satisfactorily in yellow pine as oxygen diffuses through the wood. By the substitution of acetic acid and arsenite ions, Paris green was precipitated progressively as the solution lost acetic acid, poles being successfully treated with this solution at 30°. Useful results were obtained from a solution containing copper sulphate 2·80%, sodium carbonate 0·75%, arsenious oxide 1·25%, sodium acetate 1·55%, sodium chloride 4·00%, and sodium bisulphite 0·70%, which gave a mixture of Scheele's green and Paris green in the wood. Zinc meta-arsenite precipitated from sulphuric acid (*d* 1·84), 0·20%, zinc sulphate 2·00%, calcium acetate 1·40%, soda ash 0·05%, and arsenious oxide 1·50% was satisfactory when chestnut and cedar poles were treated, and proved to be soluble in the acid produced from fungi. Toxicity tests show that 4 pts. of Scheele's green in 10,000 of nutrient jelly inhibits the growth of *Fomes annosus*, whilst the insoluble arsenites generally have a toxicity towards this fungus which is several times greater than that of zinc chloride. Boiling tests show that these arsenites are highly resistant to leaching effects.

III. Basic substances which do not contain poisonous ions were investigated as wood preservatives because the wood-rotting fungus *Fomes annosus* produced in 14 days acid equivalent to 113 mg. of acetic acid. Compared with the corresponding chlorides, sodium and barium carbonates are 16 times as toxic towards *Fomes annosus*, presumably because of their alkalinity, whilst the carbonates of strontium and calcium are, respectively, 3 and 5 times as toxic as the corresponding chlorides. The hydroxide and oxychloride of zinc are as toxic as zinc chloride although less soluble. Sodium carbonate lacks permanence in rain, but barium carbonate, although about half as toxic as zinc chloride, is worthy of test as a wood preservative because of its non-corrosive nature, low electrical conductivity, and permanence to weather. W. G. Carey.

Use of shale oil as a wood preservative. A. M. Sowder (Ind. Eng. Chem., 1927, **19**, 1180—1182).—In order to determine the value of shale oil as a wood preservative the toxicity of pyridine and quinoline towards cultures of *Fomes annosus* was investigated. The fact that pyridine is much less toxic than quinoline and has a low b.p. and high volatility renders it unimportant when judging the suitability of shale oil as a preservative. Quinoline has been shown by Weiss (J.S.C.I., 1911, **30**, 1348) to compare favourably in toxicity with cresol, phenol, and mercuric chloride; shale oil containing from 2·43—3·06% of quinoline should inhibit the growth of destructive fungi when injected into wood to the amount of 12 lb. of oil per cub. ft. of timber. W. G. Carey.

Wood preservation. Schwalbe.—See II.

Patents.

Manufacture of hydraulic cements. L. Forsén (E.P. 272,163, 19.11.26. U.S., 3.6.26).—A mixture of Portland cement, burnt lime, and a granular material is ground until the Portland cement, under the action of the granular material, has reached such a high state of sub-division that it reacts quantitatively with water, and the product is separated from the coarser particles by blast separation. The lime, with water, converts into colloidal solution any finely-ground granular material and, if this contains silica, hardening calcium silicates are formed. W. G. Carey.

Manufacture of fused cement and gases containing phosphorus. W. Kyber (E.P. 256,622, 4.8.26. Ger., 4.8.25).—A mixture of a material having a high content of alumina, *e.g.*, bauxite, and a phosphate is melted with sufficient carbonaceous material to reduce the phosphorus. W. G. Carey.

Production of fused cement. Studienges. f. Nutzbarmachung Schweizerischer Erzlagerstätten (Swiss P. 117,718, 4.7.25).—Mixtures of blast-furnace slag with quicklime are heated in an electrical induction furnace. The setting time of the cement is reduced by rapidly cooling the product. L. A. Coles.

Impregnation of wood. Montan, Inc., Assees. of J. R. Coolidge (E.P. 265,206, 26.1.27. U.S., 26.1.26. Cf. U.S.P. 1,556,570; B., 1925, 960).—Wood, fibreboard, etc. before impregnation under pressure with a wax of high m.p., *e.g.*, montan wax, is treated under pressure at a raised temperature with a material which readily penetrates the wood, is miscible with or dissolves the wax, and is toxic, *e.g.*, creosote oil or β-naphthol, after which vacuum treatment is applied to withdraw a large part of the liquid forced into the wood. W. G. Carey.

Wood preservative. Rütgerswerke-A.-G. (E.P. 266,296, 17.12.26. Ger., 20.2.26).—Sodium fluoride is added to the preservative consisting of an aqueous solution of sodium silicofluoride, to prevent iron with which the preservative comes in contact from being attacked. H. Royal-Dawson.

Manufacture of bricks and tiles. E. W. Tillberg, Assr. to S. Hellström (U.S.P. 1,647,063, 25.10.27. Appl., 17.2.27. Swed., 9.6.26).—Suitable clay is heated at 300—900°, the temperature being so adjusted that a portion of the clay still remains in a plastic condition. The product is then crushed, moistened with water, pressed into bricks and tiles, and fired without previous drying. H. Royal-Dawson.

Manufacture of bricks, blocks, etc. from materials typifiable by dolomite. F. L. Duffield (E.P. 278,120, 26.7.26).—A mixture of a flux (*e.g.*, ferric oxide, clay, or basic slag) with granular dolomite is sintered at 1200—1700° and run directly into cast-iron or

steel moulds in which it is pressed to the required shape and density, after which the product is allowed to cool.
W. G. CAREY.

Separation of solids from liquids [in sand or gravel washers]. J. SOUTHALL (E.P. 277,500, 23.9.26).

Heat-treating of materials (E.P. 269,480).—See I.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Relation between the slagging of iron and of manganese in the basic open-hearth process. E. FAUST (Arch. Eisenhüttenwesen, 1927, **1**, 119—126; Stahl u. Eisen, 1927, **47**, 1871—1872).—The relation between the iron and manganese in the metal and slag at the end of the treatment in the open-hearth furnace is given by the equation $[Mn]/[Fe] = K(Mn)/(Fe)$, square brackets indicating the composition of the metal and round brackets that of the slag. Numerous experiments with widely varying charges and using different manganiferous materials showed that for the basic-lined furnaces the value of K was 247 within the normal working range of temperature, and for acid-lined furnaces and in the Bessemer converter, 1350.
A. R. POWELL.

Nitridation of special steels. L. GUILLET (Compt. rend., 1927, **185**, 818—821).—Steels of varying compositions were tempered, reheated, and nitrided for 90 hrs. in a current of ammonia. Physical tests then showed that the type of fragility known as "Krupp's disease" was absent from chromium–aluminium–molybdenum steels containing a little nickel, and was most and least marked for chromium–aluminium–nickel steels and chromium–aluminium steels, respectively. Ordinary chromium–aluminium–molybdenum–nickel steels showed the disease to a slight extent, but were otherwise satisfactory, and in no case was the hardness decreased by the heating at 500° for 90 hrs.
J. GRANT.

Rapid determination of manganese in steels. A. QUARTAROLI (Annali Chim. Appl., 1927, **17**, 375—378).—A small quantity of the metal is dissolved in nitric acid, most of the latter being then expelled and the liquid diluted with water so that the solution contains 1 g. of the steel per 5 litres. If the proportion of manganese is fairly high, this liquid is further diluted twentyfold, and 20 c.c. of it are treated at 20° with 4 drops of ammonia solution (d 0·95), 0·5 g. of powdered glass, and 2 c.c. of hydrogen peroxide solution capable of giving about 22 c.c. of oxygen at 20° when completely decomposed. The liquid is kept at 20°, and is shaken at intervals of 5 min., the volume of oxygen liberated being read. In absence of manganese the volumes of oxygen after 5, 10, 15, 20, and 25 min. are 0·1, 0·3, 0·5, 0·7, and 0·8 c.c.; if 12 pts. of manganese are present per 100 pts. of iron, the volumes are 0·6, 1·4, 2·3, 3·6 and 4·5. Values are also given for 11, 10, 9, and 8 pts. of manganese, the volumes for the last amount being 0·3, 0·9, 1·4, 1·8, and 2·4 c.c. If the steel is comparatively poor in manganese, 100 c.c. of the solution containing 1 g. of iron per 5 litres are heated rapidly to boiling with 0·25 g. of sodium acetate and filtered, the determination being carried out as above on 20 c.c. of the filtrate. The presence of the acetate modifies the result, and a table is given showing the results for different percentages of manganese, but the latter have been accidentally omitted from the table.
T. H. POPE.

Rapid determination of the silicon content of ferrosilicon by means of the density. M. VON SCHWARZ (Chem.-Ztg., 1927, **51**, 815).—A curve is given showing the relation between the density of ferrosilicon at room temperature and its content of silicon. The density is taken as the average of ten determinations on different portions of each sample, either in solid or in powder form, in order to obviate any slight irregularities in composition. In the absence of impurities or of pores in the material, the silicon content can be determined with an accuracy of 1%.
F. R. ENNOS.

Determination of sulphur in ores. K. K. JÄRVINEN (Z. anal. Chem., 1927, **72**, 81—100).—The sources of error in sampling and dissolving the ore and in precipitating the sulphuric acid by means of barium chloride or benzidine hydrochloride are discussed in detail, and the following method is recommended for the determination of sulphur in pyrites and other sulphide minerals. The finely-powdered sample (0·5 g.) is warmed on the water-bath with 10—15 c.c. of a mixture of 2 vols of nitric acid (d 1·4), 2 vols. of hydrochloric acid (d 1·19), and 7 vols. of water. After 15—30 min. the solution is cooled, and any black specks of impure sulphur are dissolved by shaking with 1 c.c. of bromine and 1—2 c.c. of ether; finally the excess of ether and bromine is expelled by gentle warming, and the solution evaporated to dryness. The residue is evaporated again with 5 c.c. of hydrochloric acid (d 1·1) and then digested with dilute hydrochloric acid until all soluble compounds are dissolved. After removing gangue matter by filtration the clear solution is diluted to 150 c.c., treated with 2 c.c. of 10% hydroxylamine hydrochloride solution, heated to boiling, and treated with 60 c.c. of cold 0·25N-barium chloride added drop by drop over a period of 5 min., followed by a further 10 c.c. added at once. The precipitate is collected immediately on a close paper, washed with hot water, ignited wet, and weighed; a correction factor of 3·3 mg. is added for every 1 g. of barium sulphate obtained. Equally good results are obtained by precipitation with 0·3N-benzidine hydrochloride solution from a volume of 300 c.c. after reducing the iron as above, evaporating to 100 c.c., cooling, and filtering. The precipitate is washed with cold water, transferred to the precipitating vessel, boiled with 150 c.c. of water, and titrated with sodium hydroxide (free from carbonate), using phenolphthalein as indicator.
A. R. POWELL.

Electrolytic deposits of cadmium for the protection of metals and alloys against corrosion. J. COURNOT and J. BARY (Compt. rend., 1927, **185**, 773—774; cf. B., 1927, 489).—Cadmium deposits on copper are superior to those of nickel or zinc in that they are adherent, non-permeable, and resistant to the corrosive effect of a salt-water fog, an exposure of 1 hr. of which is equivalent to two weeks in the normal atmosphere. On the other hand they lack hardness, and as a result rapidly lose their fine finish. Double-deposition experi-

ments using cadmium–nickel, copper–nickel, and zinc–nickel combinations showed that the presence of cadmium and of zinc produced the most and least satisfactory results respectively, the cadmium in particular producing a fine finish and a close deposit. J. Grant.

Corrosion of lead by mineral waters. P. Kaja (Z. angew. Chem., 1927, **40**, 1167—1168).—The lead covering of a telephone cable was found to be completely corroded away over a short length, principally on the under side. The cable was encrusted with calcium carbonate and the lead converted into carbonate and oxide. A short distance away a mineral spring containing calcium carbonate and sodium chloride in large quantities was found. Although mineral waters may be conveyed through lead pipes without corrosion taking place, conditions are different when the water is in free contact with the oxygen of the soil. The chlorides present appear not to have taken any appreciable part in corrosion. C. Irwin.

Determination of traces of certain impurities in lead. B. S. Evans (Analyst, 1927, **52**, 565—572).—*Antimony.* The method recommended is a modification of that used for determining antimony in copper (B., 1922, 144 A). The lead nitrate solution is precipitated with sulphuric acid, evaporated until acid fumes are evolved, taken up with hydrochloric acid, and sodium hypophosphite added, and, after boiling, benzene. The filtrate is boiled with cleaned copper foil, the copper washed, covered with cold water, and sodium peroxide added. After 5 min. the liquid is poured off and zinc sulphide added to it and the washings. This liquid now contains all the antimony. Sulphur dioxide is passed into the acidified filtrate and gum arabic added, and after making up to volume the colour is compared with a standard. *Arsenic* is determined by a similar process and is dissolved in a measured excess of $0 \cdot 01N$-iodine. *Tin.* If 1—2% is present the sample is dissolved in hydrochloric acid and bromine, tin reduced to the stannous state and dissolved, and the solution titrated with iodine, but for small quantities use is made of the very different solubilities of lead in hot and cold aqueous media. Dissolution is effected in a percolator (A., 1926, 707) carrying an upright condenser, and so arranged that the lead chloride is continuously removed. Concentrated hydrochoric acid, potassium iodide, and platinum foil are now added to the tin solution, granulated zinc is dropped in, and the solution boiled in a current of carbon dioxide and eventually titrated with iodine in absence of air. *Bismuth.* Lead chloride is precipitated and to the filtrate, reduced in bulk, sulphuric acid is added, lead sulphate removed, the filtrate evaporated, dissolved in water, and the bismuth determined in the cooled solution colorimetrically with potassium iodide. *Sulphur.* The sample is digested with nitric and hydrochloric acids, evaporated to dryness, hydrochloric acid and hot water are added, and the solution is boiled. Sulphates are precipitated with barium chloride in the usual way, the sulphur present being calculated from the weight of precipitate obtained. D. G. Hewer.

Thermal conductivity of light alloys. C. Grard and J. Villey (Compt. rend., 1927, **185**, 856—858).—Thermal, mechanical, and electrical data of a number of alloys of aluminium with copper or silicon are given. Additions of copper to aluminium and magnesium gradually decrease and increase, respectively, their thermal and electrical conductivities till these become constant and almost equal for the two types of alloy. The ratios of the thermal and electrical conductivities confirm the Wiedemann–Franz law, and the former increase very slightly between 80° and 150°. Magnesium with 4% Cu (hammered) has thermal and mechanical properties superior to those of the alloy containing 12% Cu, and has a lower density. J. Grant.

Control of soldered joints by magnetic spectra. A. Roux (Compt. rend., 1927, **185**, 859—861).—A perfect soldered joint gives the same magnetic spectrum (with continuous lines of force) as the steel of which its two portions are composed, whilst a faulty joint may be detected by means of the characteristic results obtained when the lines of force produced by the poles of a U-shaped electromagnet, placed symmetrically across the line of solder, are rendered visible by means of iron filings. J. Grant.

Electrolytic recovery of zinc from pyrites calcination residues rich or poor in copper. H. Paweck and H. Wenzl (Z. angew. Chem., 1927, **40**, 1106—1112).—In the Kellner process (Austr. P. 5484 of 1901) the residues are treated with sulphur dioxide, giving a solution of zinc, copper, and iron bisulphites, from which zinc sulphite is precipitated by boiling out of contact with air. The zinc sulphite is collected on a filter, and converted into sulphate by air oxidation, its solution being freed from iron by treatment with bleaching powder and zinc oxide, and is then further converted into zinc chloride with sodium chloride. The solution of zinc chloride is finally electrolysed. This process is modified by Paweck in that the zinc sulphite is converted into sulphate by the calculated quantity of warm 10% sulphuric acid, and the resulting solution is electrolysed in a Paweck diaphragm cell. These processes apply, however, only in the absence of appreciable quantities of copper, zinc sulphite being obtained free from copper only when the solution contains less than $0 \cdot 3\%$ Cu and more than $1 \cdot 6\%$ Zn. The yield of zinc sulphite increases with increasing concentration of bisulphite, and attains 75% with a concentration of $6 \cdot 5\%$ Zn. With residues rich in copper the precipitation of zinc sulphite is interfered with, and the copper must first be removed by heating the bisulphite solution to 75°, when the copper is precipitated as sulphide, the zinc remaining in solution. Traces of copper sulphate in the solution are removed by adding zinc, and the remaining sulphites of iron and zinc are oxidised by adding bleaching powder and blowing in air. Addition of zinc dust rich in oxide precipitates ferric hydroxide, and the clear zinc sulphate solution is electrolysed, with a lead anode and steel cathode. With residues rich in zinc and poor in copper the zinc sulphite is converted into sulphate by addition of the anolyte (containing 10—12% of sulphuric acid) from a previous electrolysis of zinc sulphate. W. T. K. Braunholtz.

Patents.

Manufacture of steel in open-hearth furnaces. W. and H. Mathesius (E.P. 258,835, 28.6.26. Ger., 22.9.25).—Malleable scrap iron alone, or with the

addition of up to 20% of pig iron or its substitutes, is smelted and the temperature raised to 1900° or over; a material free from oxide is produced, and the usual boiling process can be omitted. H. ROYAL-DAWSON.

Production of [smelting charges of] coke and [iron] ore masses. R. TORMIN (E.P. 265,228, 28.1.27. Ger., 29.1.26).—Ground coal, coke, lime, etc. are intimately mixed with fine iron ore and degassed out of contact with air in separate retorts, closed on all sides, but not gas-tight. H. ROYAL-DAWSON.

Treatment of low-carbon steel and iron. F. KRUPP A.-G. (E.P. 274,016, 2.9.26. Ger., 7.7.26).—Low-carbon steel or iron is quenched from above 650° and afterwards drawn at temperatures up to 750°. Material so treated is more resistant to ageing influences and to the effects of liquids and vapours. C. A. KING.

Method and apparatus for refining steel and pig iron. J. MAXIMOFF, M. S. DE COSTA, and R. P. D. KREBS (E.P. 262,136, 27.11.26. Fr., 28.11.25).—Steel or pig iron is refined by means of centrifugal force in a heated rotary drum, the speed of rotation then being reduced and the collected impurities removed by gravity through a central orifice. The drum may be heated internally by gas or electricity and cooling may be effected by a current of air passing through tubes arranged within the drum and connected to annular distributing chambers. C. A. KING.

Casting of [steel] ingots. (SIR) C. A. PARSONS and H. M. DUNCAN (E.P. 278,032, 29.4.26).—Molten steel is cast in moulds having greater longitudinal than vertical dimensions. The bottom of the mould is a chill surface, and the sides are covered with refractory material of some thickness, the whole being heated to a high temperature before casting. Electrical heating units may be incorporated in the sides of the mould to assist in keeping the upper part of the ingot molten until the final stage of solidification. C. A. KING.

Method of annealing or heat-treating steel or other metals. F. W. GUIBERT (U.S.P. 1,644,828, 11.10.27. Appl., 24.10.25).—The metal is heated to the required temperature and then quenched, being maintained in a vacuum during the entire process. M. E. NOTTAGE.

Production of sheets, bands, wires, etc. from ferrosilicon alloys for the improvement of their electrical properties. O. H. DÖHNER (E.P. 276,953, 22.4.27. Ger., 4.9.26).—For the production of an alloy having a low "watt loss," high-silicon iron is cold-worked within critical deformation limits and reheated to the recrystallisation temperature (600—900°) determined by the composition of the alloy. C. A. KING.

Rustless alloy steel. J. T. WHITELEY, Assr. to E. J. SPINKA (U.S.P. 1,647,491, 1.11.27. Appl., 19.7.26).—The alloy contains 8·75% Cr, 3% Ni, 1% Cu, 1·5% Si, 0·2% C, 0·75% Zr, 0·5% Ti, and the remainder iron. F. G. CROSSE.

Erosion-resistant ferrous alloy. C. B. JACOBS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,647,096, 25.10.27. Appl., 11.1.23).—The alloy consists of 3—15% Mn, 1—5% Cr, at least 2% C, and the remainder iron. F. G. CROSSE.

Preparation of low-carbon alloys. V. B. BROWNE (U.S.P. 1,645,126, 11.10.27. Appl., 9.1.23. Renewed 24.2.27).—A metal bath is heated by means of a carbon arc and alloying compounds are introduced, the absorption of carbon being prevented by means of an oxidising slag. M. E. NOTTAGE.

Treatment of machine parts made of austenitic manganese steel. BÖHLER GEBR. & Co., A.-G., Assees. of J. FUCHS (Austr. P. 106,326, 15.10.25).—Articles of manganese steel containing 0·5—1·4% C and 10—17% Mn are heated in a carbonising medium for several hours at 800—1100°, allowed to cool slowly, reheated to 900—1000°, and quenched in oil or water. The resistance to wear is considerably increased by this treatment. A. R. POWELL.

Mechanical ore-roasting furnace. MANUF. DE PROD. CHIM. DU NORD ETABL. KUHLMANN (Austr. P. 105,797, 7.7.24. Fr., 29.8.23).—A mechanical roasting furnace comprises a number of superimposed hearths alternately stationary and rotating, the latter being carried on rollers running on cirular rails; both types are provided with water-cooled, rotating, metallic, toothed rabbles. A. R. POWELL.

Tunnel annealing furnace. R. SCHUBERT and C. PLETSCH (E.P. 277,541, 26.1.27).—To ensure uniform heating the flared orifices of the burners are provided only in the bed and side walls of the tunnel, and those in the same wall are so directed that the currents of hot gases intersect, thus minimising the tendency of the gases to rise. H. HOLMES.

Continuous roasting furnace for zinc or lead ores. L. TOCCO and M. LANDI (F.P. 619,660, 4.6.26).—A continuously operating, rotating, roasting furnace is provided with one or more separately revolving parts so as to allow of charging and discharging. A. R. POWELL.

Alloys and their application to the manufacture of electrical conductors. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (E.P. 278,454, 10.7. and 24.9.26).—A quaternary alloy of high electrical resistance and high permeability contains 0—17% Fe, 1—5% Al, 1—5% of a fourth element, *e.g.*, chromium, the remainder being nickel. A more particular composition shows 8—11% Fe, 2—3% Al, 2—3% Cr, the balance (not less than 84%) being nickel. C. A. KING.

Thermostatic metal. C. E. FRY, Assr. to H. A. WILSON Co. (U.S.P. 1,643,809, 27.9.27. Appl., 31.5.21).—A strip of Monel metal is fused to a similar strip of invar steel to give a couple, of which the shape alters with change of temperature. T. S. WHEELER.

Production of metallised surfaces on bodies containing sulphur. A. I. G. WARREN, and PRECIOUS METAL INDUSTRIES, LTD. (E.P. 278,437, 7.7.26. Addn. to E.P. 196,063; B., 1923, 565A).—Materials, *e.g.*, tarry or bituminous substances, containing sulphur are placed in contact with metal or a metallic compound and heated until a film of metallic sulphide is formed. After removal of the excess metal the sulphide is reduced to metallic form. C. A. KING.

Coating of metals with aluminium or magnesium or aluminium or magnesium alloy. R. HOPFELT

and C. P. Nolden (E.P. 278,415, 1.7.26).—A coat of aluminium or magnesium or their alloys is applied to the surface of a metal in the presence of silica or siliceous material, and the whole is then heated at 800—1200°. The coating metal may be used in the form of powder mixed with silica, kaolin, and an organic binder to form a paste which may be brushed or sprayed on to the base metal. C. A. King.

Production of a weatherproof oxide layer on electron metal. W. Pieper, Assr. to J. B. Soellner Nachf. Reisszeugfabr. A.-G. (U.S.P. 1,642,309, 13.9.27. Appl., 22.5.24. Ger., 4.7.23).—Electron metal (a manganese–zinc–aluminium alloy) is immersed in sodium hydroxide solution containing potassium nitrate for 20 min. at 110°. T. S. Wheeler.

Improving the mechanical properties of aluminium containing magnesium. G. Giulini (Austr. P. 106,021, 29.7.20).—Aluminium containing 0·5—1% Si and 0·2—1% Fe is alloyed with 0·1—0·5% Mg and the cast alloy is annealed for 1 hr. at 540—560°, quenched, and tempered at 160—170° for 10 hrs. The Brinell hardness of the alloy so treated is 90—100, and the tensile strength 3—3·5 tons/cm.^{2} after rolling or drawing, compared with 35—45 and 1·4—1·8 tons/cm.^{2} in the case of unalloyed aluminium. A. R. Powell.

Manufacture of a [non-tarnishing] silver-silicon alloy. M. G. Corson, formerly Korsunsky, Assr. to Electro Metallurgical Co. (U.S.P. 1,643,304, 27.9.27. Appl., 16.5.24).—The alloy comprises silver together with 1·5—6% Si and 1—50% Cd, Al, Zn, Sn, or Sb. T. S. Wheeler.

Bearing metal. S. Deiches (Austr. P. 106,211, 14.5.25).—An alloy for use as a bearing metal comprises 30—64% Pb, 10—15% Cu, and 30—40% Sb, with the addition of small quantities of cadmium, mercury, or arsenic. A. R. Powell.

Bearing metal. P. Tutzky (Austr. P. 105,701, 15.5.25).—A bearing metal alloy contains 61·5% Pb, 13·5% Sb, 20·5% Sn, 2·5% Cu, 1% Ni, and 1% Mo. A. R. Powell.

Solder for aluminium and its alloys. Stuttgarter Versicherungs-Ges.m.b.H. (Swiss P. 118,501, 13.8.25. Ger., 15.6.25).—A solder for aluminium contains 30—70% Al together with tin, zinc, and small quantities of other metals. *E.g.*, it may contain 45 pts. of aluminium, 37 pts. of tin, 18 pts. of zinc, 2 pts. of copper, 1·5 pts. of silicon, 1 pt. of lithium, and 0·5 pt. of iron. Equal weights of copper and aluminium are first melted together, the iron and silicon added, followed by the remainder of the aluminium and the zinc, tin, and lithium; finally the molten alloy is deoxidised with ferric chloride, skimmed, and cast into sticks. A. R. Powell.

Uniting dissimilar metals. H. C. Mougey, Assr. to Gen. Motors Research Corp. (U.S.P. 1,644,741, 11.10.27. Appl., 2.1.23).—Articles of flexible copper and of a ferrous material may be united by placing between them a flexible brass sheet coated with zinc and heating it sufficiently to cause the brass and zinc to alloy. M. E. Nottage.

Flux for welding and soldering. H. Theurer (Austr. P. 105,680, 14.8.25).—A flux for use in brazing iron and steel and in the autogenous welding of cast iron, copper, and brass comprises an alcoholic solution of ethyl borate containing naphtha and an emulsifying agent, such as sulphonated castor oil. A. R. Powell.

Alloy for pen points. K. K. Ledig, Assr. to Amer. Platinum Works (U.S.P. 1,647,301, 1.11.27. Appl., 1.3.26).—The alloy contains 60% Mo, 10% W, 10% Ru, together with 20% of a nickel–copper alloy consisting of 70% Ni and 30% Cu. F. G. Crosse.

Recovery of aluminium from oily metallic mixtures etc. J. G. G. Frost, Assr. to Nat. Smelting Co. (U.S.P. 1,646,239, 18.10.27. Appl., 17.3.26).—After subjecting the oily mixture of finely-divided aluminium and iron to the action of a magnetic separator, the organic matter is burned in an oxidising flame, and the metallic residue again separated. H. Royal-Dawson.

Manufacture of metal polishes. F. E. Genge (E.P. 278,078, 29.6.26).—Pyridine is used as part of the cleansing agent, with one or more of the abrasives in general use. H. Royal-Dawson.

Pickling of metals. J. H. Gravell (E.P. 278,398, 29.4.26).—See U.S.P. 1,632,833; B., 1927, 785.

Open-hearth furnace. G. L. Danforth, jun., Assr. to Open Hearth Combustion Co. (U.S.P. 1,647,213, 1.11.27. Appl., 21.1.21).—See E.P. 183,247; B., 1922, 764 a.

Roasting of metallic ores. H. S. Mackay (U.S.P. 1,647,050, 25.10.27. Appl., 23.4.23. U.K., 19.5.22).—See E.P. 205,528; B., 1923, 1230 a.

[Conveyor for use in] metallurgical operations [employing briquettes]. New Jersey Zinc Co., Assees. of F. G. Breyer and E. H. Bunce (E.P. 268,302, 15.2.27. U.S., 25.3.26).

Method and apparatus for degreasing metal etc. J. Savage (E.P. 278,061, 23.6.26).

Recovery of lead from ores (E.P. 278,093).—See VII.

XI.—ELECTROTECHNICS.

Mercurous sulphate electrode for testing storage batteries. S. Makio (Amer. Electrochem. Soc., April, 1928. Advance copy. 7 pp.).—From measurements at temperatures between 11° and 28° the single potential of the normal mercurous sulphate electrode, $Hg|Hg_2SO_4,N\text{-}H_2SO_4$, is found to be given by $\varepsilon_h = 0{\cdot}676 - 0{\cdot}00026(t - 18)$ volt at $t°$. Measurements have been made of the single potentials of the positive and negative plates of a lead accumulator during charge and discharge. H. J. T. Ellingham.

Explosion phenomena in the coating of mirrors—a warning. W. Meyer (Chem.-Ztg., 1927, **51**, 804).—Explosions occurring in the silvering of mirrors are attributed to the formation at the liquid margins of silver amide and silver nitride ("fulminating silver") through the use of too concentrated solutions of ammoniacal silver nitrate and potassium hydroxide. The avoidance of over-production, the exclusion as far as possible of all organic impurities by lightly covering the reaction vessels, and the bringing into solution, by gradual addition of ammonia, of the brownish-black

specks of silver oxide precipitated on addition of the potassium hydroxide are further precautions advocated.
R. BRIGHTMAN.

Light sources for testing dyes. HADFIELD.—See VI.

Refractories for electric furnaces. KOTHNY.—See VIII.

Electrolytic protection of metals. COURNOT and BARY. **Corrosion of lead on cables.** KAJA.—See X.

Electrical properties of rubber-sulphur compounds. CURTIS and others.—See XIV.

Electrometric determination of ash in sugar. ŠANDERA.—See XVII.

PATENTS.

Electrolyser diaphragms of the filter-press type. R. PECHKRANZ (E.P. 271,043, 16.3.27. Switz., 14.5.26).—The diaphragm is mounted in a frame of circular, elliptical, or polygonal contour with no internal angle of 90° or less. A single metal member cast in one piece or bent to shape and soldered at its ends may be used.
H. HOLMES.

Electric furnace. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of D. F. NEWMAN and C. STEENSTRUP (E.P. 265,950, 29.1.27. U.S., 15.2.26).—Means are provided for inserting or withdrawing a portion of the furnace charge through the same normally-closed opening without the remainder of the charge in the furnace being exposed to the atmosphere.
J. S. G. THOMAS.

Electric furnace. F. A. J. FITZGERALD, Assr. to HARPER ELECTRIC FURNACE CORP. (U.S.P. 1,646,058, 18.10.27. Appl., 26.5.25).—The lining of the furnace chamber is made of coalescing carborundum blocks.
J. S. G. THOMAS.

[Electric] furnace. A. N. OTIS, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,646,213, 18.10.27. Appl., 16.7.25).—An arm supported at one side of the furnace can swing over the furnace, whilst a second arm pivoted on the first and extending lengthwise thereof is connected with the furnace cover, which is lifted by a lever pivoted on the end of the first arm and acting on the second.
J. S. G. THOMAS.

Electric furnace. J. A. SEEDE, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,646,221, 18.10.27. Appl., 23.3.25).—A pair of adjustable electrodes extend in arcing relation into an enclosed space enclosed by refractory walls in the furnace chamber and surrounded by the charge.
J. S. G. THOMAS.

Plates for electric accumulators. G. FROMONT (E.P. 250,985, 20.4.26. Fr., 20.4.25).—Chemically pure salts of alkali, alkaline-earth, and earth metals, produced by the action of sulphuric acid on bicarbonates or carbonates of the corresponding metals, are incorporated in the formation electrolyte and in the paste for filling the plates.
J. S. G. THOMAS.

Discharge electrodes for electrical gas-purifying apparatus. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 442,644, 22.6.24).—The electron-emitting properties of the usual metals or alloys used for the construction of the electrodes, such as copper, bronze, brass, nickel, and tungsten, are improved by the addition of about 0·3—3·0% Th, or of one or more of the alkaline-earth metals, magnesium, or the rare-earth metals.
L. A. COLES.

Treatment of hydrocarbons (F.P. 615,581).—See II.

Flame-tinted glass (E.P. 271,481). **Resistant stoneware** (U.S.P. 1,642,754).—See VIII.

Alloys for electrical conductors (E.P. 278,454).—See X.

White lead (E.P. 277,723).—See XIII.

XII.—FATS; OILS; WAXES.

Linoleic acid content of bone grease. I. H. STADLINGER and E. TSCHIRCH (Chem.-Ztg., 1927, 51, 667—669; cf. Kaufmann, B., 1926, 165, 758).—Free cyanogen displaces iodine from iodides, is displaced from thiocyanates by bromine, and behaves in addition and substitution reactions like a halogen element. New titration methods for oils and fats are based on these facts, the thiocyanogen being used in solution in an indifferent solvent. So-called "rhodanometric" determinations of the iodine values of fats and oils have been made and compared with Hanus iodine values. In compounds with one ethylenic linking the results were the same by either method; when two such linkings were present the "rhodanometric" iodine value was only half the theoretical, *e.g.*, linoleic acid, iodine value 181·09, gave a "rhodanometric" value 90·545. Details are given for the preparation of the reagent and its titration with 0·1*N*-thiosulphate solution. The authors find that the solution is not constant when compared with other volumetric solutions, and tabulated results of its titre confirm this. Polymerisation sets in after some days, and it is not recommended to use a solution of the technical reagent after 8 days. The determination of linoleic acid in fats by Kaufmann's rhodanometric method should be followed by determination of the Hanus iodine value.
H. M. LANGTON.

Linoleic acid content of bone grease. II, III. H. STADLINGER and E. TSCHIRCH (Chem.-Ztg., 1927, 51, 686—688, 706—708).—II. In the thiocyanate determination of the iodine value an 80% excess of thiocyanate and 16—24 hrs.' treatment was found to be sufficient. The proportions of oleic, linoleic, and saturated fatty acids are calculated from the Hanus iodine value and the thiocyanate iodine value. The highest linoleic acid content of a trade sample of the acid, determined by this method, was 82·1%. A number of samples of bone fat extracted from fresh or cooked beef bones averaged 5·2% of linoleic acid. A series of mixtures of the two were prepared. This could not be done by melting them together as a precipitate appeared, and analysis of such mixtures gave discordant results. With cold-mixing, however, the determined linoleic acid contents were within 1% of the quantity taken and the accuracy of the method was confirmed.

III. A large number of trade samples of bone grease were examined for Hanus iodine value and thiocyanate iodine value, and the linoleic acid content was deduced. Certain discrepancies led to the conclusion that a preliminary acid treatment was essential to the securing of reliable results. Taking only acid-treated samples, the mean figure for "refined bone grease" was 8·9% of linoleic acid and for "crude bone grease" 7·3%.

This compares with 5·2% of linoleic acid in laboratory-prepared bone grease, and the cause of the difference requires further research. C. Irwin.

Changes in the characteristic indices of fats during the production of rancidity. K. Täufel and J. Cerezo (Anal. Fís. Quím., 1927, **25**, 349—362).—The development of rancidity in fats is of two types, viz., the ketonic or aromatic type as in the case of coconut oil, and the oleic acid type, as in the case of unsaturated glycerides. Data obtained for a twenty years' old sample of colza oil, by the usual methods for characterising fats, are given and are discussed from the point of view of the authors' theories.

G. W. Robinson.

Determination of the purity of olive oils. C. Milani (Annali Chim. Appl., 1927, **17**, 389—390).—When 5—6 c.c. of olive oil are shaken in a test-tube with 1 c.c. of a 1% solution of eosin in acetone, a pale pink coloration develops which disappears either almost immediately or when the tube is heated in a water-bath. Under similar treatment, a seed-oil, such as sesamé, cottonseed, almond, castor, etc., gives at once a red or sulphur-red coloration, which tends to become deeper, even when the tube is heated. Olive oil adulterated with a seed-oil also gives the permanent red or pink coloration. This test is disturbed by the presence of water or other extraneous substance. T. H. Pope.

Fruits of Siberian cedar and the cedar oil. W. Rutschkin (J. Oil Fat Ind., Russ., 1926, No. 4—5, 13—16; Chem. Zentr., 1927, I, 2783).—The seeds of the Siberian cedar (*Pinus cembra* L.) contain about 60% of a light yellow drying oil (*d* 0·93, *n* 1·485, saponif. value about 192, iodine value (Hübl) 130—160, Reichert–Meissl value 0·44—3·77) which is edible and can also be used for the preparation of a slow-drying varnish. E. H. Sharples.

Patents.

Extraction of oil from vegetable seeds etc. C. Downs and R. A. Bellwood (E.P. 278,145, 23.8.26).—Vegetable seeds etc., travelling in the form of a thin layer of meal along an endless band of porous material in an enclosed chamber, are alternately sprayed with a suitable extracting solvent and subjected to the action of an evacuating device whereby oil and solvent are withdrawn into sumps situated beneath the conveyor band. S. S. Woolf.

Extraction of oil from the blubber of marine animals. A./S. Forsøksdrift (E.P. 257,582, 3.8.26. Norw., 31.8.25).—Whale blubber etc. is finely ground and a substantial proportion of the water present is removed forthwith by heating *in vacuo*. The oil is then extracted from the tissue residuum by pressure.

S. S. Woolf.

Treatment of wax. Paint remover containing wax. O. E. Enell, Assr. to Chadeloid Chemical Co. (U.S.P. 1,646,280—1, 18.10.27. Appl., [a, b] 24.12.24). —(a) A normally solid animal or vegetable wax is purified by treatment with hot trisodium phosphate solution. (b) A paint remover comprises suitable solvents and treated wax (*v.s.*). The wax is softer, has lower sp. gr., and tends less to granulation and separation from the solvents than untreated wax of like kind.

S. S. Woolf.

Hydrogenation process (G.P. 441,164).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Constitution of artificial ultramarines. II. Ultramarine-blue with high silica content and silver, silver–sodium, and silver–selenium ultramarines. F. M. Jaeger and F. A. van Melle (Proc. K. Akad. Wetensch. Amsterdam, 1927, **30**, 479—498; cf. A., 1927, 715).—The variations in the *X*-ray powder spectrogram of ultramarine-blue Guimet No. 7553, as silver is substituted for sodium, giving finally 46·5% Ag, have been investigated. For comparison, several other silver compounds were prepared, *e.g.*, from ultramarine-pink Guimet OVR, giving 34·4% Ag; ultramarine-blue No. 13 of the Vereinigte Ultramarinfabriken, 34·2%; and ultramarine-green No. X from the same source, 51·0%. These silver ultramarines give practically identical spectrograms. The replacement of sodium by silver results in a diminution of the constant a_0 of the fundamental lattice from 9·06Å. to 8·93Å., but the original periodic structure is preserved. The relative intensities of corresponding interference images in the spectrograms of the sodium- and the silver-ultramarines, respectively, are, however, appreciably modified. The substitution of sulphur by selenium appears to leave the general structure unchanged. The general character of the relative intensities of corresponding diffraction images and their sequence also appear to remain the same, with a few exceptions. The film of the selenium derivative, however, is more clouded, probably due to the greater diffracting power of the selenium as compared with the sulphur atoms. The lattice constant $a_0 = 9·10$Å. of the selenium derivative is only very slightly greater than that of the sulphur derivative. Similar behaviour is observed when selenium substitutes sulphur in silver-ultramarine. The results suggest that the sodium and silver atoms take a more important part in the formation of the "rigid" periodic structure of the ultramarines than the sulphur or selenium atoms, which may be statistically dispersed through the whole structure. Rotation spectrograms of the minerals nosean and hauyne have also been obtained, in order to confirm previous conclusions as to the fundamental grating and space-groups of these compounds. In addition, the mineral analcite has been examined, but, although closely related chemically to the other silicates studied, its structure appears to be quite different. M. S. Burr.

Testing of nitrocellulose lacquers. E. von Mühlendahl and H. Schulz (Z. angew. Chem., 1927, **40**, 1185—1187).—In the Karussell test the samples are mounted on a vertical wheel which makes one revolution per 15 min. During a revolution they are immersed in water, then exposed in turn to radiant heat, giving a temperature of 60—70°, and to the rays of a mercury arc lamp. It is shown that one day in this test produces results closely parallel to one month of weathering. Another test consists in producing a film of lacquer of known thickness on a mirror, removing it by softening it with water, drying, and determining on a series of

days the breaking strain and breaking elasticity of the film. Increase of softening medium in the lacquer reduces the initial strength, but gives a more elastic film with a longer life. Another method of determining elasticity consists in bending sheets of known thickness and breadth through an angle of 180° around rods of different diameters. The rod which causes fracture gives a measure of the elasticity. These tests are all somewhat unsatisfactory, but taken in combination together with practical experience they give important indications of what improvements are desirable.

C. Irwin.

Rôle of solvents and diluents in varnishes. B. Scheifele (Farben-Ztg., 1927, **33**, 207—209).—A dissertation on the structure of varnishes and lacquers and their films from the colloidal point of view, the significance of "solvate sheaths" as transition regions between pure disperse phase and pure dispersion medium being stressed. The original size and structure of the disperse phase and the solvent power of the solvent are factors influencing the formation of such sheaths and the resulting viscosity changes. Mechanical properties of films are not necessarily enhanced by complete interpenetration of disperse phase and dispersion medium. Even when drying oil and similar films are excluded, film formation cannot be regarded as the reverse of the dissolution process, effects due to vapour pressure, latent heat of evaporation, surface tension, and the formation of adsorbed layers being superimposed. S. S. Woolf.

Damar Penak. R. W. Blair and F. E. Byron (Malayan Forest Rec. No. 4, 1926, **3**, 1—12).—The chemical and physical properties of 5 grades of Damar Penak (pale, yellow, amber, dust coarse, and dust fine) and 3 grades of "dead" (opaque) Damar, all obtained from *Balanocarpus Heimii*, are tabulated and compared with the results of earlier observers. The acid value of pale Damar Penak increases with age, light or absence thereof having no effect on this change. Details of varnish trials and of methods of purification of the resin are given. S. S. Woolf.

Cedar oil. Rutschkin.—See XII.

Patents.

Manufacture of white lead by electrolysis. R. S. Carreras (E.P. 277,723, 17.5.26).—In the manufacture of white lead in a single-chamber tank by the electrolysis of, *e.g.*, sodium chlorate solution saturated with carbon dioxide, exhausted electrolyte is run off and fresh regenerated electrolyte is supplied continuously, the degree of saturation and the amount of electrolyte being kept constant. S. S. Woolf.

Manufacture of zinc oxide. New Jersey Zinc Co., Assees. of J. A. Singmaster, F. G. Breyer, and E. H. Bunce (E.P. 268,301, 12.2.27. U.S., 27.3.26).—A charge of zinciferous and carbonaceous material is heated under substantially non-oxidising conditions, and the metallic zinc so formed meets a blast of relatively cool oxidising gas. The stream of gases, containing in suspension fine zinc oxide particles at about 700°, passes to an appropriate collecting room, being maintained at a temperature not below 125° during the collection of zinc oxide and separation of exhaust gases. Deposition of sulphur compounds on the pigment is thereby inhibited.

S. S. Woolf.

Application and treatment of varnish. G. R. Clover, Assr. to Cooper Hewitt Electric Co. (U.S.P. 1,646,010, 18.10.27. Appl., 18.7.23).—Varnished articles conveyed through a tunnel chamber are exposed to heat and to light rich in ultra-violet rays. S. S. Woolf.

Coating compositions. Brit. Thomson-Houston Co., Ltd., Assees. of W. P. Davey (E.P. 257,243, 28.7.26. U.S., 24.8.25).—The tendency to scumming in "water japans" consisting of an aqueous emulsion of asphaltic material, oleaginous material, and rubber latex, is inhibited by the addition of lamp black, clay, or other loading material of suitable density. S. S. Woolf.

Application of coatings of nitrocellulose lacquers. Wolff & Co., and H. I. Schulz (E.P. 262,440, 1.12.26. Ger., 4.12.25).—Priming coats for cellulose lacquers are based on nitrocellulose of higher nitrogen content and lower ether–alcohol solubility than the nitrocellulose used in the subsequent lacquer coats (which may differ progressively among themselves in these respects). Guncotton nitrocellulose containing more than 12·6% N and incompletely soluble in ether–alcohol is preferred for use in primers. S. S. Woolf.

Manufacture of condensation products of carbamide and formaldehyde. H. Barthélemy, Assr. to Soc. Ind. des Mat. Plastiques (U.S.P. 1,645,848, 18.10.27. Appl., 23.11.25. Fr., 16.12.24).—An alkaline-earth carbonate is added to a mixture of carbamide and formaldehyde and the whole is boiled. S. S. Woolf.

Manufacture of a synthetic resin [shellac substitute]. J. McIntosh and E. Y. Wolford, Assrs. to Diamond State Fibre Co. (U.S.P. 1,642,078—9, 13.9.27. Appl., [a] 12.8.20; [b] 20.2.25).—(a) Equal parts of a phenol and glycerol are heated, first under reflux with a small quantity of sulphuric acid for 6—8 hrs. and then with hexamethylenetetramine at 125° for 24 hrs. (b) A phenol and glycerol are condensed without addition of hexamethylenetetramine. T. S. Wheeler.

Production of purified rosin. W. B. Logan, Assr. to Acme Products Co., Inc. (U.S.P. 1,643,276, 20.9.27. Appl., 21.4.26).—Wood rosin is heated at 300° for 2 hrs. to render it more suitable as a substitute for gum rosin.

T. S. Wheeler.

Nitrocellulose for lacquers (E.P. 252,382).—See V.

Treatment of lead chloride (E.P. 278,093).—See VII.

Paint remover containing wax (U.S.P. 1,646,281).—See XII.

Resin from rubber (U.S.P. 1,642,018).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Density and electrical properties of the system, rubber–sulphur. I. Density of rubber–sulphur compounds. A. T. McPherson. **II. Electrical properties of rubber–sulphur compounds.** H. L. Curtis, A. T. McPherson, and A. H. Scott (U.S. Bur. Stand., Sci. Paper No. 560, 1927, **22**, 383—397, 398—418).—Vulcanisation of rubber with sulphur is accompanied by a distinct increase in density; a maximum increase is observed at 19% of combined sulphur, which corresponds with the formation of a compound $(C_5H_8)_2S$. The dielectric constant and the

power factor curves show minimum values at this composition, whilst the curves showing variation in resistivity and in coefficient of thermal expansion with percentage of combined sulphur also undergo a marked inflexion. The changes in density accompanying vulcanisation are not simple, one complicating factor being the transformation of sulphur into allotropic forms. As the proportion of combined sulphur in rubber is increased from 0 to 32% the dielectric constant increases to a maximum at 10·5%, decreases to 19%, and then increases slightly up to 32%. The power factor behaves similarly except that the maximum occurs at 13% of combined sulphur; the resistivity increases up to 26% and then decreases up to 32%, inflexions occurring in the curve at 10·5, 13·5, and 19%. These results indicate the possibility of three additional compounds, viz. $(C_5H_8)_4S$, $(C_5H_8)_3S$, and $(C_5H_8)_4S_3$. The electrical breakdown is affected to such an extent by circumstances of the test that no definite relations can be indicated with the proportion of sulphur. The effect of free sulphur on the electrical properties of rubber (vulcanised or not) is very small relative to the effect of combined sulphur. D. F. TWISS.

PATENTS.

Production of vulcanised rubber and accelerators therefor. H. W. ELLEY and D. H. POWERS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,643,205, 20.9.27. Appl., 5.3.25).—A *p*-nitrosoaryldialkylamine, *e.g.*, *p*-nitrosodimethylaniline, is heated at about 100° for 1—2 hrs. with 0·5—2 mols. of a primary, secondary, or tertiary aliphatic or heterocyclic amine, *e.g.*, *n*-butylamine or pyridine, with or without addition of a solvent, *e.g.*, ethyl alcohol, to give a product of value as an accelerator. T. S. WHEELER.

Vulcanisation of rubber articles and apparatus therefor. NEW LIVERPOOL RUBBER CO., LTD., and F. AMENDE (E.P. 278,064, 26.6.26).—In the vulcanisation of rubber articles, *e.g.*, shoes of rubber or with rubber soles, in an atmosphere of compressed air or other gas, the gas is circulated inside the vulcanising chamber and is preheated before admission, thereby ensuring a more even temperature than is obtained with the use of internal heating coils only. D. F. TWISS.

Method of reclaiming rubber and fabric from scrap. HERCULES POWDER CO., Assees. of L. T. SMITH (E.P. 274,797, 25.9.26. U.S., 20.7.26).—Scrap rubber is heated at from 100° to 180° with a solvent consisting mainly of dipentene. This solvent may be the fraction b.p. 160—200°, d^{15} 0·86, from the distillation of pine wood or may consist of chemically dehydrated pine oil. The resulting solution is filtered to remove fabric and fillers, and is then evaporated for the recovery of the rubber. By heating with caustic soda under pressure used solvent may be rendered fit for re-use.
D. F. TWISS.

Manufacture of a rubber conversion product [resin]. H. L. FISHER, Assr. to B. F. GOODRICH CO. (U.S.P. 1,642,018, 13.9.27. Appl., 28.12.26).—Rubber is heated with trichloroacetic acid at 100° for 2 days, with or without addition of a solvent, *e.g.*, benzene, and a phenol. T. S. WHEELER.

XV.—LEATHER; GLUE.

Bactericidal action [in tanning processes] of chromium salts and its general origin. S. HILPERT, L. PANETH, and E. SCHLUMBERGER (Z. angew. Chem., 1927, **40**, 1086—1089).—Two factors must be considered separately, viz., the direct action of acidity on bacteria, and the influence of acidity on the action of chromium salts on protein. With *Staphylococci* the action of acidity persists up to p_H 3, and with *B. coli* up to p_H 2; beyond this the action of the chromium salt supervenes. With *Staphylococci* and a 0·01% solution of chrome alum there is a feeble bactericidal action up to p_H 4, due to acidity. At p_H 4·29 the reaction between the chromium salt and protein begins, and reaches a pronounced maximum at p_H 4·46. As neutrality is more nearly approached this action disappears again. *B. coli* gives a maximum at the same p_H value, but is more resistant to acidity. Commercial green chromium sulphate, which is a stronger tanning agent than chrome alum, exerts a more powerful bactericidal action at the optimum p_H value than the latter. W. T. K. BRAUNHOLTZ.

PATENTS.

Treatment [tanning] of animal tissue. F. W. WEBER (U.S.P. 1,642,054, 13.9.27. Appl., 19.3.25. Renewed 31.1.27).—Prepared hides, after treatment with a solution of soap, *e.g.*, sodium stearate, are immersed in a solution containing a rare-earth chloride, or a mixture of such compounds, cerium chloride excepted, sodium chloride, and, preferably, zinc sulphate. They are then treated with sodium phosphate solution.
T. S. WHEELER.

Treatment of hides and skins preliminary to tanning. R. BOTSON (U.S.P. 1,647,953, 1.11.27. Appl., 21.5.27. Belg., 2.6.26).—See E.P. 272,195; B., 1927, 758.

Synthetic tanning agents (G.P. 441,432).—See XX.

XVI.—AGRICULTURE.

Phenomena of absorption by soil colloids, studied by means of manganese salts. A. QUARTAROLI (Annali Chim. Appl., 1927, **17**, 384—389).—When a small quantity (1 g.) of fine soil is left in contact, with occasional shaking, with 100 c.c. of a solution of a manganese salt containing 1 pt. of manganese per 50,000 of water at the ordinary temperature, appreciable proportions of the salt are absorbed. This absorption is not an ordinary precipitation in stoicheiometric proportions. Subsequent treatment of the soil with successive quantities of water shows that part of the manganese salt is readily given up, whereas part is held tenaciously either as absorption compounds not easily decomposed by water or as true insoluble compounds.
T. H. POPE.

Influence of soil treatment on the assimilability of nutrients, in the seedling method of Neubauer. P. HAUSCHILD (Z. Pflanz. Düng., 1927, **10A**, 37—53).—Drying increases the apparent amount of available nutrients in soils, as determined by the Neubauer method. The effect is greater as the temperature of drying increases. The possibility that changes in the proportion of calcium bicarbonate present in soils under

different conditions affect the solubility of phosphates is discussed. Drying may also assist in the decomposition of native phosphate. A. G. POLLARD.

Decomposition of hydrogen peroxide by soils. K. SCHARRER (Biochem. Z., 1927, **189**, 125—149)—In the gas-volumetric determination of the power of a soil to decompose hydrogen peroxide, neutral peroxide must be used, as the catalytic power of the soil, although not a direct function of its p_H, is much greater in neutral or alkaline than in acid soils. The greater the manganese, iron, and total calcium content of the soil, the greater is its activity. Sandy soil has the least and loam and clay soil the greatest activity. Soils containing humus give, usually, higher values than mineral soils of similar composition, and high moor soils, on account of their acidic character, give smaller values than the more alkaline lower moor soils. The loss on ignition of soils involves loss of catalytic power only in so far as it reduces the carbonate content, and thereby the alkalinity of the soil. No relationship was detected between the hygroscopic nature of the soil and its activity. When a soil is sieved, the coarser grain shows less activity than the finer. Treatment of the soil with hydrochloric acid, aluminium chloride, mercuric chloride, or copper sulphate decreases, and with sodium hydroxide increases its activity. Treatment with potassium cyanide also increases its activity by virtue of its alkalinity. Boiling and heating the soil at 100° until it becomes constant in weight decreases its activity, whilst ignition results in considerable loss of activity except in moor soils rich in iron oxide. The activity is also decreased by irradiation with ultra-violet light. Manuring the soil only affects the catalytic power in so far as it alters its reaction. The soil containing the lowest number of bacteria possessed also the lowest catalytic activity, but there was no direct relationship between bacterial count and activity. Manganese compounds have the greatest catalytic power, and iron oxide comes next. P. W. CLUTTERBUCK.

Movement of water in soils and subsoils. A. F. LEBEDEV (Z. Pflanz. Düng., 1927, **10A**, 1—36).—Experimental work is described showing the mechanism of the transference of water from one soil horizon to another, and, in particular, the accumulation of subsoil water. The movement of soil water may result from the influence of gravitational and capillary forces or by the distillation of "hygroscopic" moisture brought about by changes in temperature and humidity at varying depths from the soil surface. Factors controlling these water movements, their limitations and effectiveness in soils and subsoils of different types are discussed in the light of existing theory and of practical results obtained.
A. G. POLLARD.

Neubauer method for determining the solubility of rock phosphates [in soils]. R. RAUSCHER (Forts. Landw., 1927, **2**, 85; Bied. Zentr., 1927, **56**, 395—397).—In experiments with Gafsa phosphate, the total intake of phosphorus by the seedlings increased with the amount of phosphate added, but the percentage utilisation of the added phosphate decreased. Fineness of grinding was of great importance in determining the solubility of rock phosphates, but the relative effect of fineness on solubility was specific to each type of phosphate. The method does not offer a common basis of comparison for rock phosphates. The fertiliser value of these can only be ascertained by individual experiment. A. G. POLLARD.

New method for the ready determination of manganese in soil. A. QUARTAROLI (Annali Chim. Appl., 1927, **17**, 379—383).—Two grams of the soil are treated with boiling hydrochloric acid, most of which is then expelled, and the solution made up to 250 c.c. In 100 c.c. of this liquid the iron is determined approximately by precipitating with ammonia in presence of ammonium chloride, and washing the precipitate successively with boiling water, boiling 10% sodium hydroxide, and water. Part of the original 250 c.c. is then diluted to contain 1 g. of iron in 20 litres, 100 c.c. being used for the determination of the manganese by the method employed for steel (B., 1927, 910). If the proportion of manganese is very small, the hydrochloric acid solution of 2 g. of the soil is rendered almost neutral, and the iron, aluminium, and phosphoric acid are precipitated with sodium acetate. The liquid is made up to known volume and filtered, one half of the filtrate being concentrated to a small volume on a water-bath, treated with bromine water, and again concentrated to expel the bromine. The manganese hydroxide is collected on a small filter and dissolved in hydrochloric acid, the solution being evaporated to dryness and the residue made up in water to 100 c.c., or, for a very small amount of the precipitate, to 20 c.c. The catalytic determination of the manganese is then carried out on 20 c.c. of the solution. If only the readily assimilable manganese is required, 2 g. of the soil are treated with 2 c.c. of acetic acid on a water-bath until effervescence ceases, the liquid being filtered, the filter washed, and the filtrate evaporated to dryness on a water-bath. With a calcareous soil, the residue is moistened several times with hydrochloric acid and evaporated to dryness to expel most of the acetic acid. Finally the residue is made up to 250 c.c. with water, and 20 c.c. are used for determining the manganese catalytically. A table is given which shows the volumes of oxygen evolved for solutions containing from 1 g. of manganese in 125,000 to 1 g. in 4,000,000 c.c. T. H. POPE.

Colorimetric determination of potassium in aqueous extracts of soils as an indicator of lack of manuring. A. NĚMEC (Biochem. Z., 1927, **189**, 50—56).—A colorimetric method for the determination of potassium, depending on the brown colour obtained with potassium chloroplatinate and zinc chloride in hydrochloric acid solution, is described and used to demonstrate that an approximately direct relationship exists between the potassium content of aqueous extracts of a large number of soils of different sources and the amount of potassium in these soils assimilable by plants. The method can be used to determine the potassium supply of a soil and to separate soils rich from those poor in assimilable potassium.
P. W. CLUTTERBUCK.

Napier grass. W. G. FRIEDEMANN (Georgia Agric. Exp. Sta. Press Bull., 1927, No. 257).—Values for immature and mature Napier grass (*Pennisetum purpureum*),

respectively, are: moisture 90·16, 85·83; ash 1·54, 1·67; crude protein 2·02, 1·30; crude fibre 2·96, 5·41; crude fat 0·14, 0·29; nitrogen-free extract 3·18, 5·50%.

CHEMICAL ABSTRACTS.

Portable calomel electrode for the determination of p_H values in the field. P. KAMERMAN (J.S. African Chem. Inst., 1927, **10**, 32—35).—In the portable calomel electrode described, special devices to prevent the contact of the calomel with the platinum are detailed. Trials showed that the instrument was reliable for the determination of the p_H value of soils, and gave results within 0·1 of those obtained by using a standard instrument.

R. A. PRATT.

Utilisation of sulphite-cellulose waste liquor for manurial purposes. J. GÖRBING (Papier-Fabr., 1927, **25**, 573—575, 633—638, 653—658, 671—673; cf. Bokorny, B., 1919, 218; 1920, 167A).—The possibility of making use of sulphite-cellulose waste liquor as a fertiliser has been investigated. The preliminary difficulty of preparing a product which could be used under agricultural conditions was finally overcome by mixing the partly concentrated liquor with ground quicklime and peat dust. *E.g.*, 40 kg. of quicklime and 11 kg. of peat dust fixed 220 kg. of waste liquor (*d* 1·21) and yielded a readily-distributable powder containing about 23% of organic matter. The detailed results of a large number of experiments with this material on a great variety of crops and soils, both in pots and in the field, are given. In almost all the experiments the cellulose manure was either without effect or actually reduced the yield and caused injury to the plants. The favourable results previously obtained by Bokorny could not be confirmed.

C. T. GIMINGHAM.

Application of Ridsdale's modification of Pemberton's method for the volumetric determination of phosphoric anhydride to fertilisers. A. M. CAMERON and W. T. DOW (Analyst, 1927, **52**, 576—580).—After destruction of any appreciable amounts of organic matter a weighed quantity of the sample is boiled with 100—150 c.c. of water and 20—30 c.c. of concentrated nitric acid, and the liquid decanted. The residue is heated with 8 c.c. of nitric acid and 5 c.c. of concentrated hydrochloric acid until evolution of oxides of nitrogen and chlorine ceases, the diluted solution is added to the decanted liquid, and the whole filtered. Sodium hydroxide is added to 25 c.c. of filtrate till a permanent precipitate is just obtained, which is redissolved by adding, drop by drop, nitric acid (*d* 1·2), followed by a further 4 c.c. of acid, a few drops of 5% permanganate solution, and, to the warm solution, one No. 7 Analoid tablet (ammonium nitrate 1·75 g., ammonium chloride 1·6 g., ammonium oxalate 0·25 g.). The resulting solution is diluted to 60 c.c. and boiled, a mixture of 25 c.c. of standard molybdate solution and 20 c.c. of water is added, and the whole shaken. After 10 min. the liquid is decanted, the precipitate washed with 0·1% potassium nitrate solution ("Lavol") into a Nessler tube of about 65 c.c., which is placed in a Leffmann–Beam centrifuge and whirled for 1 min. The supernatant liquid is decanted through a cotton-wool plug, the Nessler tube nearly filled with wash liquor, closed with a stopper, and shaken. The tube is then filled up with "Lavol," again centrifuged, and the process repeated until the precipitate is free from acid. To the precipitate, cotton-wool plug, and washings is added an excess of 0·5*N*-sodium hydroxide, and when dissolution of the yellow precipitate is complete the excess alkali is titrated with 0·5*N*-nitric acid against phenolphthalein. Water-soluble phosphate is determined on 25 c.c. (10—15 c.c. if superphosphate) of the aqueous solution obtained by the official method by just producing a precipitate with the sodium hydroxide solution and proceeding as above, and citric-soluble phosphates by adding 6 c.c. of nitric acid (*d* 1·2) to 25 c.c. of solution, boiling for 5 min., and completing oxidation by adding three No. 4 Analoids (potassium permanganate 0·25 g.) followed by a No. 7 Analoid, and, after diluting to 60 c.c., completing as before. Results agreed well with those obtained by the official methods.

D. G. HEWER.

PATENTS.

Artificial drying of crops. B. J. OWEN (E.P. 278,474, 29.7.26).—The material to be dried is exposed to a stream of hot combustion gases and air, the former being derived preferably from liquid fuel. The combustion chamber opens into a supply duct in communication with the atmosphere at one end, and having a suction-fan at the other, and is so constructed that combustion of the fuel is complete before admixture of the gases with air drawn in by the fan. The temperature of the mixed gases is controlled by the quantity of air admitted.

C. T. GIMINGHAM.

Seed-pickling materials. I. KREIDL (Austr. P. 105,704, 18.2.25).—The materials consist of slightly soluble basic copper salts used in the dry state. *E.g.*, the seed is thoroughly mixed with finely ground basic copper chloride or sulphate so as to give an even coating on the grains.

C. T. GIMINGHAM.

[Plant] stimulant and fertiliser. I. G. FARBENIND. A.-G., Assees. of G. PFÜTZER (G.P. 443,130, 5.2.25).—Iron formate is claimed for use alone or with a fertiliser (*e.g.*, sodium nitrate) to increase crop yields.

C. T. GIMINGHAM.

Manufacture of a fungicide containing copper. J. D. JENKINS and E. F. BERGER, Assrs. to PITTSBURGH PLATE GLASS CO. (U.S.P. 1,642,370, 13.9.27. Appl., 17.11.25).—Precipitated copper hydroxide mixed with gum arabic and maize syrup is heated at 100° to give a product which forms in water a colloidal solution containing metallic copper and cuprous oxide.

T. S. WHEELER.

Copper-arsenic dust [fungicide]. G. E. SANDERS, Assr. to RICHES, PIVER & CO. (U.S.P. 1,642,511, 13.9.27. Appl., 9.8.24).—See U.S.P. 1,541,753; B., 1925, 689.

XVII.—SUGARS; STARCHES; GUMS.

Precipitation of sugar solutions with lime. Steffen process for manufacture of sugar from molasses. H. I. WATERMAN and J. S. A. J. M. VAN AKEN (J.S.C.I., 1927, **46**, 411—413 T).—Experiments involving the precipitation of sucrose solutions with increasing quantities of lime show that the phenomena observed are of a colloid-chemical character, and correspond with the view that colloidal solubility in presence of a peptiser depends

on the quantity of colloid undissolved (cf. Ostwald; also von Buzágh, A., 1927, 310), and the existence of definite compounds between sucrose and lime, *e.g.*, the so-called calcium trisaccharate, is open to doubt. A sugar solution after treatment with lime was filtered at 0°, and the filtrate at room temperature gave a precipitate of a gel character, transparent under the microscope, when air was excluded. For each mol. of sugar only 1·8 mols. of calcium oxide were removed from the solution (cf. Dedek and Tchechov, B., 1926, 927; Aten, van Ginneken, and Engelhard, B., 1927, 23).

W. J. Powell.

Deterioration of cane juices and its prevention. J. H. Haldane (Int. Sugar J., 1927, **29**, 367—370).—Mixed mill juice, treated with 2, 4, 6, 8, and 10 c.c. per litre of a solution of "E.C." (electrolytic chlorine) containing 2% of available chlorine, showed after 6 hrs. a decrease of purity of 1·08, 0·57, 0·20, 0·14, and 0, respectively, whereas the untreated sample indicated one of 3·49. After a "wash-down," and periodically during milling operations, the mill beds, mill cheeks, gutters, intermediate carriers, etc. should be sprayed with "E.C." containing 2% of available chlorine. Further, a continuous trickle of the antiseptic, diluted 1 : 500 at the beginning of the season, and increased to 1 : 200 towards the end of it, should be maintained in all the juice gutters as a preventative against deterioration.

J. P. Ogilvie.

Amino-acids and related compounds in sugar products. J. A. Ambler (Int. Sugar J., 1927, **29**, 382—385, 437—441).—Riffart's method (cf. B., 1922, 841 a) of determining amino-acids colorimetrically by means of ninhydrin (triketohydrindene hydrate) has been applied to follow the elimination of amino-acids in the refining of raw sugar, char being found to remove 70—80% of them. Raw sugar contains 0·025% N as amino-acids in a concentrated condition in the surface films of the crystal, whence they are readily removed by washing.

J. P. Ogilvie.

Decoloration of sugar juice by sulphuring before evaporation. L. Chaloupka (Z. Zuckerind. Czechoslov., 1927, **51**, 543—548, 554—558).—After passing through a triple-effect pressure evaporator with concentrator, the juice was found to be more coloured than when working under vacuum, but by sulphuring the juice after the third carbonatation, previous to evaporation, so as to reduce its alkalinity from 0·03 to 0·02% CaO, and then again carbonating to 0·01%, a much better decolorising effect was obtained than in the absence of this treatment.

J. P. Ogilvie.

After-darkening of the juices [in the carbonatation process in beet factories]. R. Brada (Z. Zuckerind. Czechoslov., 1927, **51**, 548—550).—Determinations of the colour of the juice throughout clarification in a beet factory show that after the second carbonatation it is about 20% higher than at the end of the first.

J. P. Ogilvie.

Electrical determination of the ash content of sugar factory products. K. Šandera (Z. Zuckerind. Czechoslov., 1927, **51**, 603—612).—A portable form of the author's apparatus (B., 1927, 312) has been designed for use wherever A.C. of usual voltage is available, by means of which the indication is obtained photometrically and the ash content read off directly, the temperature being compensated by the introduction of a resistance. A truer idea of the salt content of sugar solutions is obtained electrically than by the incineration method, since the latter determines only inorganic matter, but the former both inorganic and organic anions.

J. P. Ogilvie.

Sachs–Le Docte *versus* Krüger method of determining sugar in the beet. A. Le Docte (Int. Sugar J., 1927, **29**, 387).—Krüger's method, in which 78 c.c. of liquid are used per 26 g. of rasped pulp, gives a result that is 0·16% lower than the truth, the sugar content of the roots being 16%. Compared with the Sachs–Le Docte procedure, it is much more liable to give rise to various errors in routine work.

J. P. Ogilvie.

Determination of dry substance in after-product syrups and molasses. J. Mikolášek (Z. Zuckerind. Czechoslov., 1927, **51**, 9—12).—Determinations carried out during the past four seasons show that results obtained refractometrically and by desiccation (on sand) approximately agreed, those found by the former being slightly the higher. On the other hand, determinations made by the pyknometer were irregular compared with the results obtained by the other two methods. It is considered that the refractometer gives results closest to the truth.

J. P. Ogilvie.

Error in polarisation [of beet sugar solutions] produced by evaporation. C. R. Kargl (Z. Zuckerind. Czechoslov., 1927, **51**, 519—523).—Normal weight solutions of raw beet sugars, clarified with 1·5 c.c. of basic lead acetate, were filtered, and the loss of weight due to evaporation was ascertained by weighing the whole apparatus. When using an ordinary stemless funnel uncovered there was an average loss of 0·05 g. in 10 min., corresponding to an increase in the polarisation of about 0·05° V., but when covered the loss was about 0·004 g.—a negligible amount. A covered filtering device, permitting more rapid filtration than the stemless funnel, whilst obviating the error due to evaporation, is described.

J. P. Ogilvie.

Adsorption [from sugar juice] in a layer of active carbon. J. Dědek and K. L. Kácl (Z. Zuckerind. Czechoslov., 1927, **51**, 523—532).—Animal charcoal was found to adsorb practically no nitrogenous matter, contrary to certain of the decolorising carbons (here designated as C, N, and SN) tested. Adsorption of calcium oxide and mineral constituents forms generally no point of difference between animal charcoal and active carbon, which latter retains relatively large amounts of ash.

J. P. Ogilvie.

Patents.

Purification of [sugar] liquors and manufacture of sugar. E. E. Battelle (U.S.P. 1,646,079, 18.10.27. Appl., 22.7.24).—The impure sugar liquor is passed through a battery of char-containing filters arranged in series, and at intervals the filter which has been longest under liquor pressure is cut out and another one containing revivified char is inserted next to the filter which has been the shortest time under liquor pressure.

F. R. Ennos.

Manufacture of syrup. W. F. Cole (U.S.P. 1,647,082, 25.10.27. Appl., 1.5.25).—Lime is added to raw wash sugar dissolved in steam, then to every 100 lb. of syrup in the mixture is added ½ oz. of salt, after which the solution is filtered through bone charcoal.

H. Royal-Dawson.

Boiling and evaporation of sugar solutions, Berten & Co., G.m.b.H. (E.P. 276,563, 31.1.27. Ger., 12.11.26).—Refined sugar is dissolved with syrup and water in a boiler and then introduced into a collecting reservoir from which the solution is pumped in turn to a succession of boilers, which latter are brought successively into communication with a source of vacuum.

J. P. Ogilvie.

Removal of the fine grain contained in syrup and molasses. Raffinerie Tirlemontoise Soc. Anon. (E.P. 276,527, 2.11.26. Ger., 20.9.26).—Syrup and molasses are freed from fine grain by employing the surface action of particles of materials, *e.g.*, kieselguhr, paper, sugar crystals, etc., exposed to high centrifugal force. These materials may be mixed before the centrifugal action with the syrup or molasses, or may be inserted into the centrifugal in which they form a layer through which the syrup or molasses must pass.

J. P. Ogilvie.

Cleaning of molasses for the manufacture of compressed yeast. R. Kusserow (U.S.P. 1,642,929, 20.9.27. Appl., 29.10.25; cf. G.P. 439,498; B., 1927, 666).—Crude molasses diluted with water is made alkaline with calcium hydroxide, and after addition of a trace of potassium permanganate is kept at 25° for 24 hrs. A culture of lactic acid bacteria active at higher temperatures is then added and the mixture is fermented at 60° for 48 hrs., calcium hydroxide being added at intervals. Finally the liquid is heated at 80°.

T. S. Wheeler.

Manufacture of a true maple flavouring product. J. W. Sale and J. B. Wilson, Assrs. to U.S.A. (U.S.P. 1,642,789, 20.9.27. Appl., 1.4.27).—Maple sap, concentrated to *d* 1·24, is treated at 80° with barium hydroxide in quantity sufficient to precipitate all the sugar present. The liquid is filtered, and the filtrate is freed from barium with sulphuric acid, concentrated to one tenth of its original volume, and re-filtered after neutralisation with calcium carbonate. It is finally concentrated to *d* 1·3.

T. S. Wheeler.

Heat-treatment of spent bone-char and similar materials [used in sugar refining]. C. W. Burroughs (E.P. 255,079, 8.7.26).—Drying, heat-treatment, and cooling are conducted on the material while it is passing vertically down as a continuous thin layer through an annular space in which it first receives a drying heat from an impervious member forming the inner wall of the space. Its moisture is given off freely through passages formed in the outer wall into a chamber forming the second wall, through which air is preferably circulated. It is then subjected to heat treatment by receiving more intense heat from the impervious member, whereupon all gaseous products evolved pass similarly into a second chamber surrounding the outer wall, through which escape products of combustion of fue or other inert gases. Finally it passes between the impervious member and a cooling jacket to an outlet.

J. P. Ogilvie.

Purification of crude starch. K. Bratring (G.P. 441,697, 16.1.24).—By treatment of starch-protein mixtures with dilute hypochlorous acid (1·6 pts. of available chlorine for 1 pt. of the starch), the protein matter is completely removed.

C. Hollins.

Extraction of juice from [sugar] cane stalks. W. H. Morgan, sen., Assr. to W. H. Morgan, jun. (U.S.P. 1,646,762, 25.10.27. Appl., 7.12.25).—See E.P. 267,235; B., 1927, 538.

Glycerin from sugar (E.P. 278,086).—See XVIII.

Oxidation of sugars to osones (G.P. 440,389).—See XX.

XVIII.—FERMENTATION INDUSTRIES.

Malt extracts. Time of saccharification and diastatic power; phenomena hindering their determination. D. Schenk (Chem.-Ztg., 1927, 51, 814—815).—The time of saccharification of a malt extract, which is the time in minutes required by 20 c.c. of a 2% solution of the malt extract to effect the complete hydrolysis of 50 c.c. of a 3% solution of arrowroot, is useful as a control in the determination of the diastatic power. Examination of the time of saccharification and diastatic power, expressed in Pollak units, of 50 malt extracts showed that a diminution in the former was accompanied by an increase in the latter throughout the series. In certain determinations the diastatic power was lower than was to be expected from the time of saccharification. This was shown to be due to the poisonous action on the diastase of traces of copper in the distilled water, the diastatic power being diminished in some instances to one half or even one third of its normal value, whilst the time of saccharification was hardly affected.

F. R. Ennos.

Patents.

Production of yeast. Aktiebolaget Bästa (F.P. 614,037, 20.3.26. Swed., 28.3. and 1.10.25).—Sulphite waste-liquors are partially neutralised by ground limestone and fermented after removal of the precipitate.

C. Ranken.

Drying of yeast. A. K. Balls (U.S.P. 1,643,047, 20.9.27. Appl., 13.12.23. Renewed 16.8.27).—Compressed yeast is mixed with pure cellulose (10%) and dried at 20—40° for 6—8 hrs.

T. S. Wheeler.

Improving the flavour of yeast. E. B. Brown, Assr. to Fleischmann Co. (U.S.P. 1,642,537, 13.9.27. Appl., 12.3.25).—Compressed yeast mixed with sugar (1 pt.) is left for 8 hrs., and the sugar is then removed by diluting the mixture with water and inducing fermentation.

T. S. Wheeler.

Production of glycerin from sugar by fermentation in an alkaline medium. K. and N. Lüdecke (E.P. 278,086, 30.6.26).—When the fermentation of the sugar in an alkaline medium is entirely or partially finished, the yeast is separated and the volatile fermentation products are distilled off. The residual mash, which contains glycerin, is again fermented after the addition

of fresh quantities of sugar. The process is repeated as often as is required (cf. E.P. 138,328; B., 1921, 57 A).
C. Ranken.

Distillation of alcohol. E. C. R. Marks. From U.S. Industrial Alcohol Co. (E.P. 278,211, 29.11.26).—In an apparatus which comprises a distilling column, an intermediate dephlegmator, and a rectifying column, high-strength alcohol, substantially free from impurities, is produced by the distillation of liquids containing alcohol and lower-boiling impurities. The liquid alcohol and alcohol vapour of the distillate are separately conveyed from the dephlegmator to the rectifying column by valved pipe-lines in such a way that any part or all of the distillate may be introduced into the rectifier with any desired ratio between the liquid and vapour so introduced. C. Ranken.

Unfermentable and bacteria-resisting alcoholic liquids. C. O. Bertin (F.P. 617,945, 21.6.26).—Wine, beer, etc. are deprived of their content of mineral substances by means of a secondary micro-organism.
C. Ranken.

Fermentation of molasses (U.S.P. 1,642,929).—See XVII.

XIX.—FOODS.

Decomposition of free and combined cystine, with special reference to certain effects produced by heating fish flesh. L. H. Almy (J. Amer. Chem. Soc., 1927, 49, 2540—2545).—Little or no hydrogen sulphide can be detected in fresh fish flesh after heating in a sealed tube at 120° for 45 min., but relatively large amounts of hydrogen sulphide are produced if the fish flesh is somewhat stale. This effect is due to the ability of the fresh flesh to destroy hydrogen sulphide, apparently by oxidation. Added cystine, as well as that present in combined form in the fresh flesh, is destroyed by heating. The presence of considerable hydrogen sulphide in canned fish therefore probably indicates a poor condition of the material when canned.
F. G. Willson.

Detection of prohibited vegetable and coal-tar colours in foodstuffs. J. R. Nicholls (Analyst, 1927, 52, 585—589).—To an ammoniacal extract of the foodstuff 1 drop of Methyl Orange is added, the solution neutralised, then acidified to about 0·01—0·02*N*, extracted with methylated ether, and the ether extract treated with successive quantities of about 5—10 c.c. of approximately 0·01*N*-sodium hydroxide solution until no more colour is removed. An equal quantity of light petroleum is next added to the methylated ether and the mixture again extracted with sodium hydroxide. Absence of colour in alkaline layers indicates absence of prohibited dyes. The petroleum-methylated ether extract is orange-red if Aurantia is present; the methylated ether is yellow with picric acid, yellow-orange with Victoria and Manchester Yellows and gamboge, and red with Aurin. Addition to the solution of 1 c.c. each of alcohol, methylated ether, and 30% sodium hydroxide solution produces on shaking a pink soda layer with Aurin and yellow ether layer with the others. Alternatively, if to 1 c.c. of solution 2 c.c. of 30% sodium hydroxide solution are added, mixed, kept for 1 min., and 1 c.c. of methylated ether and 1 c.c. of alcohol added with shaking, the ethereal solution is colourless and soda layer yellow with picric acid and pink with Aurin. By acidifying the original solution and extracting with light petroleum, a yellow ethereal solution is produced with gamboge, whilst on removing the petroleum and adding light petroleum which has been shaken with ammonia a yellow turbidity is obtained with Victoria and Manchester Yellows. Details are given of 22 supplementary and confirmatory tests.
D. G. Hewer.

Determination of benzoic acid in foods. G. W. Monier-Williams (Analyst, 1927, 52, 572—575).—The method depends on the fact that when steam containing benzoic acid vapour is passed over moist metallic magnesium in a reflux apparatus, the benzoic acid combines with the magnesium to form a soluble benzoate extractable by washing with hot water. The success of the determination depends largely on the thoroughness of the extraction. The extract is made strongly alkaline with 40% caustic soda solution, cooled to 40—50°, oxidised with a saturated aqueous solution of potassium permanganate, excess of which is destroyed by sulphur dioxide or sodium sulphite, and the precipitated manganese dioxide dissolved in sulphuric acid. This solution, after saturation with salt, is extracted with a mixture of methylated ether and light petroleum, the solvent evaporated, and the benzoic acid sublimed. The reflux apparatus used is described. Results compared well with those obtained by steam distillation, but the method was found not so generally applicable as the latter. D. G. Hewer.

Analytical study of roasted coffee berries. J. M. Clavera (Anal. Fis. Quím., 1927, 25, 369—373).—If the percentage of ash in a roasted coffee exceeds by more than 0·5% the percentage of ash in raw coffee berries, it may be concluded that more than 10% of sugar has been added in roasting. G. W. Robinson.

Patents.

Removal of undesirable foreign flavours and odours from milk, cream, and other milk products. M. B. MacDonald, Assr. to Univ. Tennessee (U.S.P. 1,644,842, 11.10.27. Appl., 23.12.26).—The milk or milk product in liquid form is mixed with a mineral oil, and the latter is afterwards removed from the treated liquid, taking with it foreign odours and flavours.
F. R. Ennos.

Treatment of butter cream. G. A. Gray and M. B. Newburger (U.S.P. 1,643,301, 27.9.27. Appl., 16.7.23).—The cream is treated with sulphur dioxide (0·03% or the equivalent amount of sulphite) prior to shipment in order to prevent fermentation, the excess of the latter being oxidised after shipment by means of hydrogen peroxide or during the pasteurising or aerating processes. The cream is then fermented and churned into butter. F. R. Ennos.

Sterilisation of cream. B. H. Webb, Assr. to U.S.A. (U.S.P. 1,646,671, 25.10.27. Appl., 16.4.27).—Cream is heated to 80° (approx.), then rendered homogeneous at 2500—3000 lb. pressure, sealed in air-tight containers at about 118°, and sterilised. H. Royal-Dawson.

Preparation of water-soluble albumin. E. M. Meyer (E.P. 260,224, 22.9.26. U.S., 20.10.25).—A

finely-granular, acid-precipitated milk casein is washed with water completely to remove acid, and agitated for a few minutes with approximately ten times its weight of water containing 3% by weight of lime. After allowing the suspended material to settle, it is separated and the translucent solution evaporated at a temperature below 52°. The partly soluble flakes thus obtained are then rendered completely soluble in water by storing for some days with finely-powdered dibasic calcium phosphate, which is afterwards separated from the flakes by sifting through a fine mesh. F. R. ENNOS.

Manufacture of dry-powdered jelly base containing pectin and sugar. H. T. LEO (U.S.P. 1,646,157, 18.10.27. Appl., 14.3.22).—A jelly base is made by mixing powdered pectin and finely-divided sugar, the proportion of the latter being varied in accordance with the jellifying capacity of the pectin and also with its ability to effect the dissolution of the pectin in the liquid into which the composition is placed. F. R. ENNOS.

Supplemental poultry food material. W. H. LAPP, Assr. to CONCENTRATE PRODUCTS Co. (U.S.P. 1,645,703, 18.10.27. Appl., 28.12.25).—A supplemental poultry food comprising organic and inorganic materials, of which the latter contains calcium and phosphate in the proportion of 6—8 pts. of calcium to 1 pt. of P_2O_5, less than 0·2% Mg, and no fluorine. F. R. ENNOS.

Production of preserves of vegetable matters of unlimited durability. A. FAITELOWITZ (U.S.P. 1,647,089, 25.10.27. Appl., 8.3.24. Ger., 29.7.21).—See E.P. 229,100; B., 1925, 296.

Freezing apparatus for use in the concentration of fruit juices. W. A. HEYMAN (E.P. 278,799, 13.7.26).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Manufacture of benzaldehyde and benzoic acid on a large scale. R. FREUND (Chem.-Ztg., 1927, **51**, 803—804).—A description of plant and process affording by the chlorination of toluene a daily production of 150—200 kg. of pure benzaldehyde and 50—100 kg. of sublimed benzoic acid. Sublimation is performed in an aluminium vessel at 160—180°. R. BRIGHTMAN.

Pharmaceutical incompatibility of phenol. D. MIGLIACCI and R. GARGIULO (Annali Chim. Appl., 1927, **17**, 402—406).—When mixed with either naphthalene or β-naphthol, phenol immediately forms a liquid mass. The system phenol-β-naphthol exhibits a eutectic point at 26°, corresponding with 78% of phenol. Below this temperature solid solutions containing 0—40% of phenol and simple crystalline mixtures containing 40—100% of phenol are formed; at higher temperatures only pasty or liquid masses are obtained. Contrary to Yamamoto's statement (A., 1908, ii, 928), the system phenol–naphthalene has the eutectic point 29°, corresponding with 80% of phenol. At lower temperatures only crystalline mixtures are formed, although pasty masses may result owing to absorption of moisture by the phenol; at higher temperatur s pasty or liquid masses are formed. Neither system forms molecular compounds. T. H. POPE.

Extraction of ergot. I. Liquid extract of ergot. W. H. LINNELL and D. G. RANDLE (Pharm. J., 1927, **119**, 423—427).—The preparation of alkaline, acid, and neutral alcoholic extracts of ergot has been examined. An alcoholic menstruum is preferable to water, but both are inefficient as extractive media and the addition of either boric acid or calcium hydroxide does not increase their activity. Alcohol (50 or 60%) acidified with not less than 0·5% of tartaric acid is the most effective solvent, and this yields a solution which contains a maximum of about 0·1% of ergotoxine in a 1 in 2 extract. It is unnecessary to prepare extracts stronger than 1 in 2 since 0·1% repres nts nearly the limit of solubility of ergotoxine. The efficiency of the extraction on continuing the percolation with unacidified dilute alcohol is very small and the labour involved is not justified. Methods of extraction adopted in nine foreign pharmacopœias are reviewed, and it is shown that with the exception of the Dutch and United States extracts all are practically inactive owing to the choice of unsuitable solvents. Defatting should precede extraction and all extracts should be 1 in 2. Projected work is outlined. E. H. SHARPLES.

Butyrates of linalol and the linalyl acetate contents of lavender oil. A. KAUFMANN and F. KJELSBERG (Perf. Ess. Oil Rec., 1927, **18**, 418—421).—Linalyl butyrate (97·86% of ester, d_{15}^{15} 0·8970, $[\alpha]_D^{20}$ −10·02°, n_D^{20} 1·4518) and *iso*butyrate (94·49% of ester, d_{15}^{15} 0·8926, $[\alpha]_D^{20}$ —11·89°, n_D^{20} 1·4487) are much more stable than is the acetate. The butyrate requires boiling for 4 hrs. and the *iso*butyrate for 7 hrs. with a small excess of 0·5N-alcoholic potassium hydroxide before saponification is complete. The rates of hydrolysis of some esters of linalol adjusted to an ester conten of about 50% (comparable with that of a good lavender oil) by addition of linalol have been examined. The results show that, owing to the low rates of hydrolysis of the two butyrates, lavender oil cannot contain large amounts of these two esters. Determinations of the linalyl butyrate and *iso*butyrate contents of lavender oils from various sources by a modification of Schimmel's fractional hydrolysis method (Gildemeister and Hoffmann, III, p. 74) show that, whereas natural lavender oil may contain these two esters, they are only present in small amounts (below 7%) and may occur combined with alcohols other than linalol, *e.g.*, geraniol. The effect of different distillation methods for the production of the oil on the linalyl acetate and butyrate content is discussed (cf. also Langlais and Goby, B., 1927, 92, 267). The presence of the methyl ether of umbelliferone in lavender oil (Pfau, *ibid.*, 571) is confirmed. E. H. SHARPLES.

Amino-acids in sugar products. AMBLER.—See XVII.

Determination of azides. COPEMAN.—See XXII.

PATENTS.

Preparation of glycols. F. H. UNTIEDT (U.S.P. 1,641,710, 6.9.27. Appl., 11.6.26).—An olefine oxide, *e.g.*, propylene oxide, is treated with an equivalent quantity of water in presence of a trace of sulphuric acid. T. S. WHEELER.

Preparation of succinic anhydride. I. G. FARBENIND. A.-G., Assees. of F. WEBEL (G.P. 441,002, 18.12.24).—Maleic anhydride vapour is passed with hydrogen over a catalyst (nickel at 180°, copper at 300°) other than platinum; or the anhydride is reduced with hydrogen in ethyl acetate in presence of a nickel catalyst at 15°.
C. HOLLINS.

Preparation of β-hydroxybutyric acid. DR. A. WACKER GES. FÜR ELEKTROCHEM. IND., M.B.H., Assees. of G. BASEL and F. KAUFLER (G.P. 441,003, 28.5.25).—Crotonic acid is heated under reflux with dilute mineral acid (sulphuric or hydrochloric acid, or aluminium sulphate), and the resulting β-hydroxybutyric acid is purified by vacuum distillation. The ethyl ester, b.p. 179—180°, and the *butyl* ester, b.p. 209—211°, are useful softening agents in varnishes. C. HOLLINS.

Manufacture of higher aliphatic acids. I. G. FARBENIND. A.-G., Assees. of R. WIETZEL and O. KÖHLER (G.P. 441,272, 26.9.24).—The oily products from the catalytic reduction of carbon oxides or from the condensation of alcohols with or without carbon monoxide, preferably freed from lower alcohols, are heated with alkali or alkaline-earth hydroxides. From the fraction of b.p. above 140° a mixture of acids $C_6H_{12}O_2$ and $C_7H_{14}O_2$ is obtained. The fraction, b.p. 160—170°, gives almost exclusively α-methyl*iso*hexoic acid, b.p. 205°. Acids $C_5H_{10}O_2$ to $C_8H_{16}O_2$ are formed from the fraction, b.p. above 120°, obtained by passing water-gas mixed with methyl and ethyl alcohols over a mixed catalyst at 450° and 200 atm. C. HOLLINS.

Preparation of organic acids [synthetic tanning agents]. I. G. FARBENIND. A.-G., Assees. of K. DAIMLER (G.P. 441,432, 14.9.20).—Fossil vegetable materials, freed or not from bitumen, or bitumens of these in so far as they are not purely hydrocarbon, are oxidised with air or oxygen in presence of nitric acid. Brown coal residues, *e.g.*, extracted with benzene, are heated at 90° in a covered vessel in a current of air, red fuming nitric acid being allowed to percolate through the mass. The waste gases are washed successively in 50% and 25% nitric acid and water, the wash-liquors being kept at about constant strength by dilution with the next weaker liquor. When samples show no further increase in solubility, the remaining nitrous gases are removed by a current of hot air. If desired, the product is freed from insoluble matter by dissolution in water. It consists of a mixture of organic acids (38% CO_2H) and not more nitrogen (2·5%) than the starting-material. There is also formed an acid mixture soluble in alkalis or alkali acetate. Ozokerite gives a water-insoluble, alkali-soluble, acid mixture, the sodium salt of which has soap-like properties. Humic acids may also be oxidised in like manner. C. HOLLINS.

Manufacture of vanillin [and azobenzene]. R. H. BOTS (U.S.P. 1,643,804—5, 27.9.27. Appl., [A] 25.1.26; [B] 25.5.26).—The product obtained by treating oil of cloves with potassium hydroxide (potassium *iso*eugenoxide) is dissolved in aniline and heated at 100° for 2 hrs. with nitrobenzene in presence of concentrated sodium hydroxide solution, to form vanillin and azobenzene. T. S. WHEELER.

Di-p-xylylguanidine. W. SCOTT, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,642,180, 13.9.27. Appl., 27.11.22).—Di-*p*-xylylthiocarbamide is converted by treatment with lead monoxide and ammonia into *di-p-xylylguanidine*, m.p. 170°. T. S. WHEELER.

Preparation of organic compounds by means of catalytic gas reactions. I. G. FARBENIND. A.-G., Assees. of A. MITTASCH, K. WINKLER, and M. PIER (G.P. 441,433, 27.9.23).—As catalysts, especially for reduction of carbon oxides, are used in addition to oxide of zinc or cadmium difficultly reducible oxygen compounds of at least two metals of the higher groups of the periodic system. Such mixed catalysts are very resistant to catalyst poisons, and when used in the reduction of carbon oxides yield alcohols almost exclusively. A mixture of 70% of hydrogen and 30% of carbon monoxide at 200 atm. passed at 400—420° over a catalyst containing zinc oxide (1—2 mols.) and chromium trioxide (1 mol.) gives nearly pure methyl alcohol. Addition of alkali hydroxides to the catalyst leads to formation of higher alcohols. Other combinations of catalysts are oxides of: zinc, chromium, and uranium or vanadium; cadmium, chromium, and tungsten; zinc, manganese, and vanadium; zinc, vanadium, and uranium; zinc, cadmium, chromium, and uranium. The metals copper, silver, lead, or thorium, or their oxides may be added, but iron compounds are to be avoided. Ethylene is reduced quantitatively to ethane when led with hydrogen at 380—400° over a mixture of zinc oxide (3 mols.), chromic oxide (2 mols.), and uranium oxide (1 mol.). The reduction of nitrobenzene to aniline, the oxidation of toluene to benzaldehyde and benzoic acid, and of methyl alcohol to formaldehyde, and the preparation of higher alcohols from alcohol, carbon monoxide, hydrogen, and nitrogen, are described. C. HOLLINS.

Hydrogenation of organic compounds. WERSCHEN-WEISSENFELSER BRAUNKOHLEN A.-G., A. FÜRTH, and G. HILDENBRAND (G.P. 441,164, 18.1.24).—The substance to be hydrogenated is passed with steam over artificially activated carbon at 300—350°. Oleic acid thus gives 68% of stearic acid, and 32% of oleic acid is returned to the process with more steam. Benzine from brown coal is completely reduced to saturated compounds after three such hydrogenations. C. HOLLINS.

Oxidation of sugars to osones. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of A. KRAISY (G.P. 440,389, 19.9.24).—From the mixture obtained by oxidation of invert sugar or lævulose with hydrogen peroxide and ferrous sulphate (G.P. 439,115; B., 1927, 619), made strongly acid with 50% sulphuric acid, the iron is precipitated by addition of the calculated quantity of potassium ferricyanide and filtered through talcum. The filtrate after removal of sulphuric acid as calcium sulphate is evaporated. The products have an improved appearance and taste. The Turnbull's blue must be adsorbed on talcum or alumina, or on silver chloride precipitated in the solution by addition of sodium chloride and silver nitrate. C. HOLLINS.

Manufacture of therapeutic agents. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 251,651, 30.4.26. Ger., 1.5.25).—

Hydrazoic acid derivatives, especially organic azides, are active hypotonics, but since their action is fugitive they are used in the form of their molecular compounds with other hypotonic and/or antispasmodic agents. *E.g.*, benzyl *p*-iodoethoxybenzoate (G.P. 412,699) and ψ-saccharin azide (benzazide-*o*-sulphonic acid) are dissolved together in acetone and the solvent is removed in a vacuum. C. Hollins.

Isolation of acridine and acridines with free *meso*-position from mixtures with other substances. Preparation of 9-cyanoacridine. E. Wirth (G.P. 440,771—2, 18.1.25).—(A) Acridine etc. is removed in soluble form as acridan-10-sulphonic acid, $C_6H_4{<}{}^{CH(SO_3H)}_{NH}{>}C_6H_4$, by treatment of high-boiling, basic tar oils with warm sodium bisulphite solution, and may be regenerated by the action of alkali on the isolated sodium salt. From 0·25 to 0·3% of acridine is thus recovered from an anthracene oil, b.p. above 300°, *d* 1·10. (B) 10-*Cyanoacridine*, m.p. 186°, is obtained by treating acridan-10-sulphonic acid with alcoholic alkali cyanide. The intermediately formed 10-*cyanoacridan* (10-*cyanodihydroacridine*) is very quickly oxidised in air to 10-cyanoacridine, which finds application in the preparation of therapeutic compounds. C. Hollins.

Manufacture of organic compounds of arsenic. R. W. E. Stickings, and May & Baker, Ltd. (E.P. 278,444, 8.7.26).—An arsinic acid of an *o*-aminophenol is treated with an α-halogenoacyl halide (chloroacetyl chloride) in presence of caustic alkali, and the product is converted separately or in the same operation into a hydroxybenz-1 : 4-oxazinearsinic acid by hot alkali. From 3-amino-4-hydroxybenzene-1-arsinic acid and chloroacetyl chloride are obtained first the 3-*chloroacetamido*-derivative, then 3-*hydroxybenz*-1 : 4-*oxazine*-6-*arsinic acid*. The 8-*acetamido*-derivative of this is prepared from 5-*amino*-3-*acetamido*-4-*hydroxybenzene*-1-*arsinic acid* (obtained by nitration and reduction of 3-acetamido-4-hydroxybenzenearsinic acid). C. Hollins.

Manufacture of protective and curative agents from glandular organs. G. Schröder (E.P. 263,155, 14.12.26. Ger., 16.12.25).—Agents for the cure of, and protection against, infectious diseases are obtained from cell suspensions from glandular organs (spleen, thymus, etc.) by keeping them in a frozen state, then thawing and filtering. The therapeutic action of the preparations is increased by inoculating them with appropriate bacteria (tuberculosis culture etc.), which are killed by the action of the preparations, while specific vaccines are produced. B. Fullman.

Manufacture of a pharmaceutical product containing arsenic. W. Schoeller and M. Gehrke, Assrs. to Chem. Fabr. auf Actien (vorm. E. Schering) (U.S.P. 1,642,830, 20.9.27. Appl., 9.8.26. Ger., 2.7.24). —See E.P. 236,563; B., 1925, 783.

Alcohols from gases containing olefines (E.P. 249,834).—See II.

Carbazides and thiocarbazides of the naphthalene series (E.P. 278,037). **Heterocyclic compounds** (E.P. 258,553). **Chlorides of *o*-hydroxycarboxylic acids of polynuclear hydrocarbons** (E.P. 278,463). **Oxidation and reduction of organic compounds** (G.P. 441,163 and 441,179).—See IV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Preparation and properties of some synthetic photohalide emulsions. E. P. Wightman, A. P. H. Trivelli, and S. E. Sheppard (J. Franklin Inst., 1927, **204**, 491—499).—A method of preparing photohalide emulsions, somewhat different from the method of Lüppo-Cramer, is described using either colloidal silver or colloidal silver sulphide as sensitiser. To remove the excess colloidal silver in the case of the former emulsion, chromic acid is used, preferably added to the emulsion before coating. This treatment results in a considerable lowering of sensitivity, which can be restored to a large extent by bathing in a very dilute solution of ammonia containing a little potassium bromide. The emulsions show in general a sensitivity extending further into the long wave-length region than emulsions prepared without colloidal silver or silver sulphide, an observation which is in accordance with those of previous workers. The spectral sensitivity of the photohalide emulsions differs from that of those sensitised with allylthiocarbamide in being more sensitive in the red and less sensitive in the green region of the spectrum. The work supports the strain and ionic deformation theories of sensitivity, but it does not exclude the halogen acceptance theory of sensitising by silver sulphide. W. Clark.

Development of silver halide-gelatin emulsions. F. Bürki (Helv. Chim. Acta, 1927, **10**, 782—784).—Although sodium hyposulphite may be used as a photographic developer and formaldehyde as an accelerator, the double compound "Rongalite" is inert. Fixers behave differently towards the "exposure nuclei" of the latent image. Exposed plates after fixing in sodium thiosulphate solution blacken on physical development, whereas exposed plates which have been treated with ammonium thiocyanate solutions or desensitisers do not. J. S. Carter.

XXII.—EXPLOSIVES; MATCHES.

Replacement of nitroglycerin in ammonium nitrate explosives. H. Kast (Z. ges. Schiess- u. Sprengstoffw., 1927, **22**, 208—213, 243—247, 279—283).—Ammonium nitrate explosives in Germany and elsewhere must contain, by law, not less than 5% of nitroglycerin in order to ensure detonation. For reasons of economy and safety in handling, it is desirable to investigate the possiblity of obtaining the same effect either by increasing the percentage of aromatic nitro-compounds, replacing nitroglycerin by nitrocellulose, or by replacing ammonium nitrate by other suppliers of oxygen such as potassium perchlorate. In the case of donarit, 8% of T.N.T. or 10% of potassium perchlorate could be present without undue sensitiveness, although initiation was less easy. When nitroglycerin is replaced by ammonium perchlorate the resulting mixtures are equal in sensitiveness to those containing potassium perchlorate. Substitution of wood meal for wheat flour yielded a less dense explosive. S. Binning.

Glycol dinitrate as basis of gelatinous blasting explosives. A. SCHMID (Z. ges. Schiess- u. Sprengstoffw., 1927, **22**, 273—276).—Attention is called to the extended use in America of glycol dinitrate as a constituent of gelatinous blasting explosives, which remain plastic when exposed to temperatures low enough to freeze the ordinary nitroglycerin explosive. Glycol is not more difficult to nitrate than glycerin and the glycol dinitrate separates quickly from the waste acid and wash-water. Owing to the toxicity of the volatile dinitrate, a continuous nitration process is recommended. Gelatinisation of nitrocotton is effected more quickly by glycol dinitrate than by nitroglycerin, so that a slow-gelatinising nitrocotton must be used or gelatinisation will be uneven. Gelatinisation should take place at 0—1·5°, when homogeneous, transparent "gelatins" will be obtained. Compared with "gelatin" obtained from nitroglycerin, those from glycol dinitrate are stickier and not so stiff, but an increase in nitrocotton and in the absorbent used subsequently removes this disadvantage. Decrease in weight of the glycol dinitrate explosive when cartridged is practically equal to that of a nitroglycerin explosive. S. BINNING.

Determination of azides. D. A. COPEMAN (J.S. African Chem. Inst., 1927, **10**, 18—22).—Existing methods are reviewed and two satisfactory methods described. In the first method, sodium azide is treated with sulphuric acid and the resulting hydrazoic acid is distilled over into a definite volume of standard caustic soda. The decrease in alkalinity is found by titration with standard acid, using phenolphthalein as indicator. In the second method a solution of ceric ammonium nitrate is allowed to act on the azide and the resulting nitrogen is collected in a nitrometer. Both methods yield results of great accuracy. R. A. PRATT.

Analysis of nitroglycerin waste acid. A. S. WEBB (J.S. African Chem. Inst., 1927, **10**, 13—14).—Sulphuric and nitrous acids are determined by titration methods, total nitrogen in the nitrometer, and the organic matter by oxidation and subsequent titration with potassium iodide and sodium thiosulphate. The method of calculating results is given. R. A. PRATT.

XXIII.—SANITATION; WATER PURIFICATION.

Use of colloids for boiler-scale prevention. E. SAUER and F. FISCHLER (Z. angew. Chem., 1927, **40**, 1176—1183, 1276—1279).—The use of boiler-scale preventives containing tannin, hydrocarbons, albuminous substances, etc. may have a scientific foundation, as it may be that such substances act as protective colloids and modify processes of crystallisation. A series of experiments was carried out on the precipitation of calcium carbonate from its acid solution by heating with and without addition of colloids. Without any addition, the rate of precipitation was found to increase with the temperature from 40° to 98°, and with the rate of stirring up to 1200 r.p.m. These factors being uniform, it was found that addition of increasing proportions of dextrin from 0·1% to 0·8% steadily reduced the rate of precipitation. At 98° precipitation was very rapid, but the presence of 0·8% of dextrin reduced the quantity of precipitate by 70%. Gum arabic had a much slighter effect, but agar-agar and gelatin were even more effective than dextrin. In the last case a thick foam formed on the surface, and such precipitate as was deposited was a non-crystalline slime which contained organic groupings from the colloid. Similar tests under a pressure of 1—10 atm. gave analogous results. With 0·8% of dextrin, up to 85% of the calcium carbonate was retained in solution; with 0·2% of agar-agar a precipitate was at first formed but redissolved. Tannin in the proportion of 0·1% also totally prevented precipitation. In all these cases the calcium in solution is in combination with organic radicals. With smaller quantities of colloid the carbonate precipitated was of a semi-colloidal nature and not coherent. C. IRWIN.

Influence of free chlorine on the elimination of manganese from water. O. WEBER (Chem.-Ztg., 1927, **51**, 794—795).—The elimination of manganese from water by aeration and filtration of the oxidised manganese compounds depends not so much on chemical action as on biological influence. Tabulated results from a waterworks in Hanover show that the manganese content is not lessened by aeration and filtration when free chlorine is added to the raw water; considerable improvement is seen when the chlorine is added after filtration, but the manganese is entirely eliminated only when unchlorinated raw water is used for washing the coke filters. A filter requires time to attain its highest efficiency, and its manganese dioxide content is important. W. G. CAREY.

Determination of the chlorine value of effluents. H. BACH and K. GLÄSER (Z. angew. Chem., 1927, **40**, 1115—1116).—"Chloramine" is used instead of potassium hypochlorite as source of chlorine. Its 0·1N-solution is stable if kept in a dark bottle. To the filtered effluent, containing a small quantity of 10% sodium hydroxide (to fix any carbon dioxide and so prevent the liberation of chlorine from the "chloramine"), is added a known volume of 0·1N-"chloramine" solution and the mixture is gently boiled under specified conditions. A small quantity of 10% potassium iodide is added, then concentrated hydrochloric acid, and, after some minutes, starch–zinc iodide solution, and the iodine is titrated with 0·1N-thiosulphate. A blank experiment must be made. A comparison of this method with the potassium permanganate and biochemical oxygen-absorption methods shows no general agreement in the results. W. T. K. BRAUNHOLTZ.

PATENTS.

Apparatus for sewage treatment by activated sludge in combination with sludge digestion. K. IMHOFF (U.S.P. 1,642,206, 13.9.27. Appl., 25.8.24; cf. G.P. 418,319; B., 1926, 470).—The sewage is aerated in a tank, into which it is introduced at the bottom. At the top it overflows into an inner settling basin from which the effluent is removed. The sludge falls to the bottom, whence a part is returned to the aeration tank, the remainder being passed to a digestion chamber. T. S. WHEELER.

Treatment [softening] of hard water. G. PETROFF and P. SHESTAKOFF (U.S.P. 1,642,594, 13.9.27. Appl., 30.12.25).—Sulphonic acids of high mol. wt., *e.g.*, a naphtholsulphonic acid, are employed. T. S. WHEELER.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DECEMBER 23, 1927.

I.—GENERAL; PLANT; MACHINERY.

Aluminium ovens for catalytic purposes. F. Fischer and H. Tropsch (Brennstoff-Chem., 1927, 8, 323—324).—For experiments requiring a uniform temperature throughout the oven, a thick-walled, cylindrical block of aluminium is used, 50 cm. long, with 8 cm. outside diameter and 2·4 cm. bore. A thermometer pocket is bored along the length of the block, a row of burners runs underneath, and heating is automatically regulated by an Ostwald thermo-regulator. The oven is surrounded by insulating brick, and may have a removable segment to enable the contents to be visible. If it is desired to maintain a catalyst tube at different temperatures along its length, the oven is composed of ten aluminium segments separated from each other by asbestos and individually heated by separate burners.
W. T. K. Braunholtz.

Stammer colorimeter. Žert.—See XVII.

Patents.

Rotary furnace. Gewerkschaft Sachtleben, and J. Kuppers (E.P. 268,308, 18.2.27. Ger., 24.3.26).—The interior lining of a rotary furnace is provided with a series of longitudinal ribs on its outer surface which engage with projecting blocks on the interior of an outer lining, thus forming a number of longitudinal heating flues. Heating is effected by a number of gas burners in a heating chamber at the end of the furnace, which chamber connects with the flues along the muffle, and from which air is excluded. Air for combustion is introduced through ducts in the lining to different points longitudinally of the heating flues, and thus enables combustion to be regulated along the furnace. By means of closure caps to the ducts the entry of air may be controlled at any particular time by rotating the furnace, such action closing or opening any of the air ducts.
C. A. King.

Rotary chemical furnace. L. Tocco and M. Landi (E.P. 278,774, 8.6.26).—The cylinder of a rotary furnace is divided into a number of compartments by means of diaphragms. Material may be charged into any compartment, and the products arising from the particular stage of the roasting process removed. Any desired compartment is heated by electrical resistances in the walls of the furnace connected to contact plates on the casing, and waste heat from the furnace is absorbed in a stationary boiler adjacent to the furnace. An example of the regulation of the furnace when roasting lead ores is given.
C. A. King.

Tunnel kiln. P. A. Meehan, Assr. to Amer. Dressler Tunnel Kilns, Inc. (U.S.P. 1,646,208, 18.10.27. Appl., 7.7.26).—A channel in the kiln chamber wall communicates with the chamber at separated points, and an injector provided with a mixing chamber is arranged adjacent to one of the points for circulating the kiln-chamber gases.
H. Holmes.

Tunnel kiln. C. Dressler, Assr. to Amer. Dressler Tunnel Kilns, Inc. (U.S.P. 1,646,279, 18.10.27. Appl., 13.6.23).—A combustion chamber within a firebox at the side of the kiln chamber communicates with the latter by a port. The walls of the port and the sides and top of the combustion chamber are provided with cooling channels through which the air for supporting the combustion is passed.
H. Holmes.

Heat exchangers. Pfaudler Co., Assees. of E. P. Nichols and U. G. Todd (E.P. 265,133, 30.9.26. U.S., 27.1.26).—A heat exchanger is constructed of a containing casing and a number of tubular heating elements which are provided with end pieces which will clamp together and form the inlet and outlet headers for the internal fluid. The tubes are also bent so that each unit may be introduced through a manhole of any ordinary size.
B. M. Venables.

Transformation of heat, more particularly applicable to refrigerators. Chicago Pneumatic Tool Co., Assees. of R. W. Davenport (E.P. 265,562, 10.1.27. U.S., 2.2.26).—A liquid, suitable for use in a closed refrigerating cycle, is composed of mutually soluble more volatile and less volatile constituents, the latter forming a lubricant, and becoming concentrated in the evaporating zone and re-diluted in the condensing zone.
B. M. Venables.

Condensing plants. A.-G. Brown, Boveri, & Cie. (E.P. 265,629, 7.2.27. Ger., 5.2.26).—In cases where there is a use for the hot circulating water from condensers, means are provided for heating the water by supplementary live steam, should the quantity of exhaust steam condensed prove insufficient.
B. M. Venables.

Pulverising mills. E. Kramer, and Hartstoff-Metall A.-G. (Hametag) (E.P. 278,997, 11.7.27).—In a mill where the material is pulverised by the interaction of eddy currents of air, stirrers are provided which sweep the inside of the casing, especially where the action of the eddy currents is least, and create mechanical whirling which causes the material to re-enter the eddy currents.
B. M. Venables.

Disintegrators. G. Porteus (E.P. 278,920, 17.12.26).—A disintegrator is arranged with screens surrounding the beaters on both sides and peripherally, and free space is left between the screens and the outer casing for the

passage of the ground material. The screens are provided on the interior with roughened cross-bars or segments to co-operate with the beaters.
B. M. VENABLES.

Apparatus for depositing [and collecting] lump and granular material. O. DOBLHOFF (E.P. [A] 264,815, and [B] 271,403, 7.1.27. Czechoslov., [A] 22.1.26, [B] 22.5.26; Addn. to [A]).—(A) A fixed conveyor discharges the material on to a second conveyor running at right angles to it, the latter being mounted on wheels so that it can be moved as a whole parallel to the first conveyor. The second conveyor discharges on to a third conveying means (preferably in duplicate) which delivers the material to the place of storage. The last (or each) conveyor is arranged so as to swivel about two axes at right angles, so that in combination with the movement of the second conveyor any point may be reached. (B) The apparatus as above described is adapted to reclaim the material by providing the third conveyor with buckets or grabs adapted to pick up the material on reversal. This conveyor is extended above the second conveyor so as to deliver thereon.
B. M. VENABLES.

Centrifugal apparatus for collecting dust, especially asbestos in course of manufacture. L. MELLERSH-JACKSON. From ASBESTOS CORP., LTD. (E.P. 278,928, 18.1.27).—Prior to entering an exhaust fan (which would break up the fibres of asbestos) the dust-laden air enters a cylindrical vessel with horizontal axis tangentially at the circumference, passes part of the way round to a collecting hopper at the lower part of the circumference, and is withdrawn axially at one end.
B. M. VENABLES.

Separation of matter of different specific gravities. H. VELTEN (U.S.P. 1,646,506, 25.10.27. Appl., 17.9.26. Ger., 28.9.25).—The mixture of granular materials is scattered in a current of air moving at constant speed along a passage provided with walls moving in the same direction and at the same speed as the air. The particles remaining suspended are discharged separately from those deposited.
H. HOLMES.

Manufacture of agglomerated masses. R. BOWEN, Assr. to SUPER COAL PROCESS CO. (U.S.P. 1,646,385, 25.10.27. Appl., 13.3.24).—A number of heterogeneous components are subjected to a mixing action, then worked to a desired consistency, and subjected to high-frequency vibrations by means having a yielding nature.
B. M. VENABLES.

Manufacture of pellets [from molten materials]. R. W. POINDEXTER, JUN., and H. J. MORGAN, Assrs. to CALIFORNIA CYANIDE CO., INC. (U.S.P. 1,647,194, 1.11.27. Appl., 5.8.26).—The material is melted and filtered and delivered in small drops upon a cool surface from which the pellets formed are removed.
H. HOLMES.

Filter press. A. M. CAPRA (E.P. 278,940, 14.2.27).—A filter of the plate-and-frame type, but with vertical axis, is arranged so that the frames are attached to a fixed vertical board or post, and the plates are attached to a board or post that slides horizontally. The upper faces alone of the filter plates are pervious, the bottoms being unperforated and forming trays which collect the filtrate and conduct it to the outlet. When the press is full the clamping pressure is released and the plates are withdrawn, thus permitting the cakes to drop through the frames to a vessel below. The operation of withdrawing the plates also scrapes the filter surfaces ready for re-use.
B. M. VENABLES.

Thickening filtering apparatus. J. B. VERNAY (E.P. 275,573, 22.6.27. Fr., 3.8.26).—A thickening filter is made to operate continuously by discharging and cleansing the filter elements in groups. An automatic valve-operating device is described.
B. M. VENABLES.

Cake-washing means for rotary vacuum filters. W. MAUSS (U.S.P. 1,646,653, 25.10.27. Appl., 14.1.27).—Wash liquid is passed continuously through the filter from a body of such liquid supported hydrostatically by the filtrant against the outgoing segment of the drum. The liquid is replenished continuously to maintain the desired circumferential extent of its contact with the drum.
H. HOLMES.

Heat-treatment of liquid and solid materials by means of hot liquids. C. F. HAMMOND and W. SHACKLETON (E.P. [A] 278,768 and [B] 278,985, 15.4.26).—(A) The hot liquid, *e.g.*, lead, is kept in circulation by means of a pump or gas lift, and the material to be treated is entrained in the downward flow of the hot liquid, and thus carried below the surface, and is then permitted to rise to the surface, baffles being provided to cause it to take a path which is more or less circuitous according to the period of heating required. The treatment may be in more stages than one if desired. (B) A submerged flame is used for the double purpose of heating and circulating the liquid, and the downward circulation tubes are formed in the shape of ejectors to assist the entrainment.
B. M. VENABLES.

Apparatus for evaporation or concentration of aqueous solutions such as sugar solution. BERTEN & CO., G.M.B.H. (E.P. 265,127, 19.7.26. Ger., 1.2.26).—The apparatus is constructed so that a number of boiling vessels may be connected in turn to a single source of vacuum. The solution is also supplied from a common source, and may be introduced through the vacuum hood of each vessel.
B. M. VENABLES.

Apparatus for electro-osmosis of liquids containing dissolved or suspended materials. ELECTRO-OSMOSE LATINE (F.P. 619,080, 20.7.26).—The liquid to be treated and one or more washing liquids flow in counter-current through a series of frames containing electrodes and porous precipitating diaphragms forming chambers.
J. S. G. THOMAS.

Removal of liquid from the interior of rotating cylinders or drums. E. A. LEATHER (E.P. 278,882, 12.10.26).—The liquid is discharged during rotation through a hollow outlet trunnion by means of a cone having its apex at the trunnion and its base within the cylinder. The interior of the cone is provided with a spiral blade or gutter arranged to convey liquid to the outlet trunnion. The base of the cone is closed, but the liquid is lifted from the circumference of the cylinder by a bucket or other means, through an aperture in the surface of the cone near the base.
B. M. VENABLES.

Apparatus for the precipitation of liquids from vapours and gases. R. Fiala (Austr. P. 106,083, 7.12.23).—The precipitators are provided with channels which open in the direction of the stream of vapour or gas to be purified, and which are arranged at the ends opposite the point of entry of the vapour.
J. S. G. Thomas.

Separation of mixtures of gases. M. Benson (E.P. 273,229, 3.8.26. U.S., 25.6.26).—In the process of separation of a mixture of gases (*e.g.*, air) by differential dissolution under high pressure, the inert insoluble gas (*e.g.*, nitrogen) is heated while still under pressure to such an extent that on subsequent expansion through a heat engine enough work will be accomplished to effect the pre-compression of the whole mixture. The gas being inert to metals, much higher temperatures may be used than if the other constituent (oxygen) were heated. B. M. Venables.

Distillation plant for alcoholic liquids. A. J. Dupont (F.P. 619,168, 21.7.26.).—The plant comprises plates with tubes surrounded by supports which constrain the liquid to flow from each plate to the one below.
J. S. G. Thomas.

Plates for distillation columns. P. Chevalet (F.P. 619,029, 17.7.26).—Plates upon which a very thin layer of liquid is traversed by a stream of very finely-divided vapour are provided with small openings fitted with small valves which permit passage of the vapour, but prevent the liquid from running down.
J. S. G. Thomas.

Fractional distillation. H. Koppers, Assr. to Koppers Development Corp. (U.S.P. 1,646,698, 25.10.27. Appl., 6.7.21. Ger., 22.4.18).—The mixture to be separated is passed continuously through a vaporising device maintained at a temperature and pressure suitable for the distillation of the ingredient of highest b.p. of the group to be eliminated, and the vapours are passed to a dephlegmator maintained at a temperature suitable for separating one fraction, the remaining fraction being condensed in a cooler at a regulated temperature. The conditions in the vaporiser and condensing apparatus are readjusted successively to deal with groups of higher b.p., the original liquid being thus completely fractionated.
C. O. Harvey.

Centrifugal separating apparatus. R. A. Sturgeon (U.S.P. 1,648,790, 8.11.27. Appl., 24.9.26. U.K., 1.10.25).—See E.P. 257,422; B., 1926, 936.

Absorption refrigerating apparatus. Electrolux, Ltd., Assees. of Platen-Munters Refrigerating System Aktiebolag (E.P. 258,580, 10.9.26. Swed., 15.9.25).

Catalytic heating apparatus. Soc. Lyonnaise des Rechauds Catalytiques (E.P. 273,668, 21.5.27. Fr., 3.7.26).

Purifying or filtering apparatus for gaseous fluids [air for internal-combustion engines]. M. C. E. Mulot (E.P. 279,000, 26.7.27. Fr., 18.6.27).

Finely-divided substances from emulsions (E.P. 278,395).—See XIV.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Reactivity of coke constituents. G. Agde and L. von Lyncker (Gas- u. Wasserfach, 1927, **70**, 1016—1019).—A Westphalian coking coal, with 7·8% of ash and 24% of volatile matter, was exhaustively extracted with benzene at 285° and 55 atm., whereby 6·0% of solid bitumen (49·5% of volatile matter) and 4·6% of oily bitumen (92·6% of volatile matter) were obtained, leaving a residual, non-coking coal with 17·5% of volatile matter. Each of these three coal constituents was carbonised at 800° and 1000°, and the reactivity of the coke was determined at 900° by passing carbon dioxide over samples contained in a boat in a porcelain tube, the unchanged carbon dioxide being absorbed in an Orsat apparatus and the volume of carbon monoxide and gas evolved by further decomposition of the cokes being measured. The temperature of initial reduction was 380—390° (the temperature at which amorphous carbon begins to reduce carbon dioxide) in all cases except that of the 1000° coke from solid bitumen, for which it was 530—540°. The reactivity of the carbonised coal residue from the benzene extraction is much higher than that of the cokes from the solid or oily bitumen, and is higher for the 800° than for the 1000° coke. The reactivity of the bitumen cokes is due mainly to soot deposited on them during carbonisation, and decreases when this has been gasified. To obviate any disturbing influence due to the gaseous decomposition products of the cokes themselves at 900°, the reactivity of the 800° cokes was also determined at 800°. The reactivity of the extracted-coal coke was greatly diminished, whilst that of the bitumen cokes sank after 2 hrs. to nil. W. T. K. Braunholtz.

Action of bacteria on coal. W. Fuchs (Brennstoff-Chem., 1927, **8**, 324—326).—A critical summary of the work of Renault, Potter, and Galle, who have shown that bacteria are capable of living on coals, where they produce a slight evolution of gas (containing 71—85% CH_4 and 5—27% CO) and a slight rise in temperature.
W. T. K. Braunholtz.

Determination of volatile matter in fuels. L. F. Dooremans and D. J. W. Kreulen (Chem. Weekblad, 1927, **24**, 562—563).—For those fuels for which losses occur by spitting in the usual determination of volatile material, more accurate figures may be obtained by heating 1 g. of the powdered fuel, covered with 10 g. of purified and previously ignited fine sand, in a platinum crucible. S. I. Levy.

Mode of occurrence of dopplerite: an unusual constituent of peat. W. Francis and F. V. Tideswell (Fuel, 1927, **6**, 516—521).—The mode of occurrence of dopplerite, a black jelly-like material, in a number of Irish peat bogs shows that it consists of peat ulmins which have been deposited from solution, or have settled in fluid form, in fissures or voids existing in the peat. No evidence of the conversion of vegetable material into dopplerite *in situ* was observed. The means whereby the ulmin solution or hydrosol was precipitated or coagulated as dopplerite is still indefinite.
A. B. Manning.

Causes of different reactivity of carbonisation products. G. Agde and H. Schmitt (Gas- u. Wasserfach, 1927, **70**, 1000—1002).—Coke is composed of substances of differing reactivity, *e.g.*, amorphous carbon, tar coke, and small proportions of graphite. Amorphous carbon, which is derived from the coal constituents not extracted by benzene under pressure, reacts appreciably with oxygen above 170° and with carbon dioxide above 385°, and is converted into graphite slowly above 1100° and rapidly above 1600°. Tar coke, the solid decomposition residue of the tar produced during carbonisation, reacts with oxygen above 370° and with carbon dioxide only above 800°, at which temperature it undergoes further decomposition with evolution of hydrogen, accompanied by shrinkage, fissuring, and gradual graphitisation. Graphite reacts with oxygen above 500° and with carbon dioxide above 800°. The reactivity of coke depends also on the magnitude and nature of the surface it presents to the reacting gases. The partial pressure of the latter is important, and may be markedly reduced by the evolution of hydrogen owing to decomposition of tar coke at high temperatures. Variations in reactivity during a determination may arise from preferential gasification of the coke constituents, or from fissuring due to shrinkage of decomposing tar coke. W. T. K. Braunholtz.

Formation of oils from ethylene and its homologues. M. Otto (Brennstoff-Chem., 1927, **8**, 321—323).—In the presence of boron fluoride, *iso*butylene polymerises vigorously to a thick, colourless oil (boiling from 200°, with about 50% below 300°). Propylene polymerises similarly but less readily. Ethylene, under pressure and in the presence of boron fluoride, polymerises at the ordinary temperature, polymerisation being much more rapid and complete if nickel is also present. Carbon monoxide, on the other hand, greatly reduces the polymerisation. The yellowish-brown oils obtained vary in properties with the experimental conditions, *e.g.*, the viscosity decreases rapidly with increasing temperature of production, and the oils obtained with the nickel catalyst are less viscous than those prepared with boron fluoride alone. Their boiling ranges vary correspondingly. The oils resemble very closely in viscosity, flash point, etc. good lubricating oils prepared from petroleum fractions. The small quantity of boron fluoride contained in the oils may be recovered and used repeatedly, and the polymerisation of successive charges of ethylene can be carried out in a continuous process. W. T. K. Braunholtz.

Apparatus for fractional distillation [of petroleum] under reduced pressure. J. C. Morrell and G. Egloff (Ind. Eng. Chem., 1927, **19**, 1292).—An improved form of the apparatus previously described (cf. J.S.C.I., 1923, 42, 188—192 t). E. Holmes.

Apparatus for vacuum distillation of lubricating and heavy petroleum oils. M. J. Gavin and A. L. Foster (U.S. Bur. Mines Rep. Invest., 1927, No. 2819, 5 pp.).

Refractories for gas retorts. Steinhoff.—See VIII.

Ignition of gas-air mixtures. Perrott and Gawthrop.—See XXII.

Patents.

Carbonisation of coal. F. Puening (E.P. 256,942, 26.7.26. U.S., 13.8.25).—The fuel is carbonised on a series of ledges projecting from the surface of a cylinder which is internally heated. The outer casing is made to rotate and any excess fuel is removed by a separate chute and returned to the feed hopper. In another modification the fuel is fed from a series of rotating discs on to the carbonising ledges, the resulting coke being removed by scrapers. A. C. Monkhouse.

Low-temperature carbonisation of coal, lignite, etc. D. Bretherick and G. J. Glossop (E.P. 277,819, 5.1.27).—The retorts consist of a series of vertical metal tubes with horizontal and vertical ribs arranged so as to reverse the flow of heating gases at each successive horizontal flue. The retorts discharge through a rotary or horizontal door into a cooling chamber having a sloping floor and water-sealed discharge valve. The carbonisation gases are withdrawn through a water-jacketed gas off-take pipe; the waste gases are collected in flues at varying heights in order to equalise the chimney pull on each retort. A. C. Monkhouse.

Cracking, catalysing, and hydrogenation of carbonaceous materials. A. E. Bianchi and G. Guardabassi (E.P. 277,404, 8.6.26, and 278,041, 27.5.26).—The materials are carbonised in troughs containing rotating shafts with stirrers; the products of distillation pass to a gas chamber in which, suspended from a screen, are a large number of small chains kept in vibration by means of the agitators below. The chains or the surfaces of the chamber are coated with a catalyst according to the nature of the products required.
A. C. Monkhouse.

Destructive hydrogenation of moist solid fuels. I. G. Farbenind. A.-G. (E.P. 262,099, 20.11.26. Ger., 26.11.25).—The fuel is mixed with a liquid hydrogenation product obtained from the fuel and compressed. Water together with a small amount of the added liquid is removed, and the remaining dehydrated pulp passes direct to the hydrogenation vessel. A. C. Monkhouse.

Regenerative coke ovens. N. V. Silica en Ovenbouw Mij. (E.P. 269,188, 7.4.27. Holl., 7.4.26).—In order to obviate the difference in the lengths of travel of the air in the respective flues of a regenerative coke oven of the twin-flue type, the air is admitted beneath the regenerators from openings placed along the middle line of the oven battery instead of from both sides of the battery. A. C. Monkhouse.

Production of a dry mixture of vaporised fuel and air. E. R. Godward (E.P. 258,222, 2.6.26. Can., 9.9.25).—The liquid fuel is vaporised by passing the vapour–air stream through a number of expanding curved passages in which the velocity of flow is diminished and which are heated at their lower ends by the engine exhaust gases. For a fuel with a higher boiling range of 232°, the temperature of the plates ranged from 177—232° at the bottom to 66—121° at the top, giving a completely vaporised mixture. A. C. Monkhouse.

Apparatus for concentrating materials such as coal. E. Deister (U.S.P. 1,644,112—3, 4.10.27. Appl., [a] 28.11.24, [b] 15.2.26).—(a) For dry concentra-

tion the coal is delivered to a differentially-reciprocated, transversely-inclined deck within which is a box for air under pressure. The surface of the deck is provided with longitudinal channels, and in one wall of each channel—that rising abruptly toward the channel next above it—are located corrugated strips forming ducts which communicate with the air box and lead laterally therefrom to the exterior. (B) For wet concentration a transversely-inclined deck with upwardly-inclined parallel launders, all in the same plane, is used, means being provided for feeding water to each launder. The sides of the launders are reduced in height at their lower ends to enable the lighter particles to pass over them for discharge at the side of the deck, and the reciprocation is such as to cause the heavier particles to travel up the launders for discharge at the end of the deck.

H. Holmes.

Manufacture of granular carbon. E. N. Bunting (U.S.P. 1,646,389, 25.10.27. Appl., 29.4.25).—Carbon granules for microphones are prepared by heating carbonaceous material gradually to not less than 500° over a period of from 10 to 100 hrs., continuing the heating at this temperature for 1—10 hrs., and finally heating for 1—10 hrs. to above 1400°. A non-oxidising atmosphere is used throughout the process.

H. Holmes.

Apparatus for completely separating gasoline from mineral oil. A. E. Pew, jun., and H. Thomas, Assrs. to Sun Oil Co. (U.S.P. 1,645,969, 18.10.27. Appl., 23.2.24. Cf. E.P. 230,821; B., 1925, 873).—A still comprises a large elongated container provided in its upper part with means for receiving oil, distributing it laterally, and conveying it in a thin layer down a succession of inclined pans for discharge into the main body of oil. Means are provided for maintaining an equal volume of oil at each side of the longitudinal centre line of each upper pan and an equal lateral distribution in the passage from pan to pan. H. Holmes.

Fractional distillation [of petroleum hydrocarbons]. N. E. Loomis and W. K. Lewis, Assrs. to Standard Development Co. (U.S.P. 1,646,619, 25.10.27. Appl., 21.7.22).—The vapours from a continuous still are passed through a fractionating zone, at least a portion of the oil passing to the still being preheated by heat exchange with vapours from this zone, and the preheated oil being introduced into an intermediate portion of the fractionating zone and allowed to pass downwards in contact with the ascending vapours. The amount of condensation is regulated by by-passing to the fractionating zone such of the feed oil as is in excess of that required in the heat interchanger, and the vapours from the partial condensation zone are condensed and collected.

C. O. Harvey.

Treatment of oil with aluminium chloride. F. W. Hall, Assr. to Texas Co. (U.S.P. 1,647,445, 1.11.27. Appl., 15.3.20).—A stream of hot oil is brought into contact with aluminium chloride vapours, and the mixture passes to a reaction chamber maintained (solely by the heat of the mixture) at a temperature adequate for cracking and distillation.

C. O. Harvey.

Purification of hydrocarbon oils containing sulphur. Standard Development Co. (E.P. 270,626, 3.9.26. U.S., 8.5.26).—Sour oils of which the sulphur content is so high as to render the cost of refining by "doctor" solution prohibitive (*e.g.*, Reagan crude) are treated with a mixture of sulphur, caustic soda solution, and lead sulphide (which is recovered and re-used), the lead present being insufficient for the conversion of more than a small proportion of the mercaptans into lead mercaptides, and the sulphur being sufficient for the conversion of the mercaptans into less volatile polysulphides which remain undecomposed in the still on subsequent distillation (preceded by acid treatment) at temperatures not substantially above 160°. Other heavy metal sulphides may be used (*e.g.*, cupric or cuprous sulphide), and, alternatively, the sulphide may be formed by passing hydrogen sulphide into an oil containing sodium plumbite, the mixture being subsequently treated with sodium sulphide. Agitation is effected by air-blowing. The distillate may be given a final light treatment with "doctor" solution.

C. O. Harvey.

Purification of waste lubricating oils. W. A. Street and H. Hey (E.P. 278,434, 6.7.26).—The waste oil is diluted with a volatile solvent (*e.g.*, petrol), and finely-divided water and solid materials are removed by treatment with a small quantity (0·5—1%) of a reagent made by the action of strong sulphuric acid on oleic acid (cf. E.P. 176,540; B., 1922, 334 A). The diluted oil is separated from the sludge formed, and treated with concentrated sulphuric acid (1—2·5% by vol.). The oil, after settling, is separated, neutralised with an excess of solid or alcoholic caustic alkali, and run into a still where the solvent is removed by heating with closed steam coils; the heavier spirit and light oils are distilled off in a current of superheated free steam at a temperature up to 200°. The hot residual lubricating oil, after being mixed with an absorbent decolorising earth (1—2%) or active carbon (0·5%), is passed through a warm filter press from which is delivered a clear bright oil of good colour, high viscosity, and high flash point. R. C. Odams.

Handling of crude oil and residuals. A. E. Miller, Assr. to Sinclair Refining Co. (U.S.P. 1,646,760, 25.10.27. Appl., 9.5.21).—Volatile constituents are removed from asphaltic petroleum oils and residues by spraying a mixture of preheated oil and neutral gas (preheated to about 343°) into the vapour space of a still containing liquid asphaltic residues (at about 343°), through which a further quantity of heated neutral gas is passed. The vapours and the asphaltic matter free from volatile constituents thus obtained are withdrawn from the still. C. O. Harvey.

Separation of coal from dirt and like foreign substances. W. H. Berrisford (U.S.P. 1,648,716, 8.11.27. Appl., 9.12.26. U.K., 11.12.25).—See E.P. 265,341; B., 1927, 625.

Carbonisation of subdivided fuel. S. McEwen, Assr. to Internat. Combustion Engineering Corp. (U.S.P. 1,648,856, 8.11.27. Appl., 11.7.21. U.K., 11.10.20).—See E.P. 169,389; B., 1921, 805 A.

Prevention of evaporation of petroleum oils. R. E. Wilson, Assr. to Standard Oil Co. (U.S.P. 1,647,424, 1.11.27. Appl., 8.10.24).

Heat treatment of materials (E.P. 278,768 and 278,985). **Fractional distillation** (U.S.P. 1,646,698). **Viscosity of oils** (E.P. 263,781).—See I. **Refractories for gas retorts** (E.P. 278,821).—See VIII. **Acetic acid from wood distillation** (E.P. 275,158).—See XX.

III.—TAR AND TAR PRODUCTS.

Removal of sulphur from commercial benzols. H. Kiemstedt (Brennstoff-Chem., 1927, **8**, 326—327).—Free sulphur in benzol is derived mainly from hydrogen sulphide reaching the crude product from the gas. It is therefore advisable to remove hydrogen sulphide as early in the process as possible, *e.g.*, the freshly condensed distillate can be washed with sodium hydroxide.

W. T. K. Braunholtz.

Reactivity of carbonisation products. Agde and Schmitt.—See II.

Wood preservation. Curtin and Bogert.—See IX.

Patents.

Cracking etc. of carbonaceous materials (E.P. 277,404 and 278,041).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Identification of dyestuffs on cellulose acetate. C. M. Keyworth (J. Soc. Dyers and Col., 1927, **43**, 343—361).—Tables are given showing the reactions on cellulose acetate silk of 220 dyestuffs, mostly not in the Colour Index, with alcohol, 5% acetic acid, dilute hydrochloric acid, 1% ammonia solution, saline sodium hydroxide, hydrosulphites B, BX, and RS (the last of which is prepared from 100 g. of hydrosulphite NF, 50 g. of Rochelle salt, 50 c.c. of sodium hydroxide of *d* 1·4, and 800 c.c. of water), titanous thiocyanate, hydrogen peroxide, "chemic," concentrated acids, and sodium hydroxide (*d* 1·25). Basic colours are stripped more or less completely with alcohol or 5% acetic acid, less readily with hydrochloric acid. Hydrosulphite RS and titanous thiocyanate also effect decolorisation, but the colour is restored with hydrogen peroxide, which is not the case with azo or anthraquinone dyestuffs. Saline sodium hydroxide decolorises dyes of the triphenylmethane group, the colour returning on acidification. Concentrated acids afford lighter shades with basic and anthraquinone dyestuffs, darker shades with azo dyestuffs. Many developed azo dyestuffs are stripped with alcohol, but the direct dyestuffs are little affected. Azo dyestuffs are little changed by 5% acetic acid; the action of dilute hydrochloric acid or saline sodium hydroxide is variable. Anthraquinone dyes are usually stripped completely with alcohol, but 5% acetic acid, dilute hydrochloric acid, or 1% ammonia solution has little action. From these tests the following dyestuffs are shown to be identical or very similar; Auramine, Setacyl Yellow AO; Cellutyl Fast Yellow AY, Alizarine Yellow WS; Cellutyl Fast Yellow AB, Anthracene Brown WLP; Cellutyl Fast Golden Yellow, Metanil Yellow Y; Cellutyl Yellow C, Citronine Y (conc.); Cellutyl Fast Orange G, Chlorazol Orange G; Duranol Orange G, Celatene Orange, Celliton Orange R; Cellutyl Fast Orange 2R, Orange II; Setacyl Red FC, Magenta, Cellutyl Fast Bright Red; Duranol Red G, Celliton Pink R; Duranol Red 2B, Celliton Fast Pink B; Cellatene Red Violet, Celliton Fast Red Violet R; Setacyl Violet MB, Methyl Violet; Cellatene Brilliant Violet B, Celliton Fast Violet B; Setacyl Violet BR, Modern Violet; Cellutyl Fast Lilac, Anthracene Blue BDG; Setacyl Turquoise Blue S, Turquoise Blue G, Cellutyl Sky Blue; Aeronal Brilliant Blue, Setacyl Sky Blue S; Cellutyl Fast Blue, Prune Blue; Cellutyl Fast Green Y, Brilliant Green Crystals, Setacyl Green B; Setacyl Green M, Malachite Green, Cellutyl Fast Green B; Setacyl Blue Green M, Methylene Green. Setacyl Scarlet G, R, and B are mixtures of Setacyl Brilliant Pink G and Setacyl Orange CR; similarly Setacyl Black G, B, and R are mixtures of Setacyl colours and Setacyl Direct Black G, B, and R mixtures of Setacyl Direct Blue R with Setacyl Reds and Yellows. The Cellatene, Duranol, Celliton, and Celliton Fast Colours are of the anthraquinone class. The Setacyl colours are all basic colours, but the Setacyl Direct, the Cibacet, and Cellit colours belong to the azo group. The S.R.A. colours contain dyestuffs of both the azo and anthraquinone classes. S.R.A. Golden Yellow X, Golden Orange I and III, Orange I and III, Red I, III, and V appear to be azo colours. The Cellutyl colours comprise basic, azo, and mordant dyestuffs, whilst the Cellitazols include a number of organic bases which are diazotised on the fibre and developed.

R. Brightman.

Evaluation of Methylene Blue. Holmes.—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Intracellular structure of the wool fibre. J. B. Speakman (J. Text. Inst., Spec. Issue, 1927, **18**, 431—453 T).—The elastic properties of the wool fibre as a whole are those of the single cell, which is assumed to consist of an elastic cell wall, enclosing a fibrillar structure the interstices of which are filled with a viscous medium. This structure tends to lie preferentially along the axis of the fibre and is heterogeneous in composition. Under small stress, extension of the cell is at first delayed by internal friction, Hooke's law being obeyed up to 2% extension for different wools in water and for the same wool at different humidities; for the same wool in water at higher temperatures (up to 100°), deviation begins between 1·4 and 2% extension. Above this critical value rapid extension occurs chiefly by rotation of the fibrillæ, the rate being determined by the viscosity of the medium within the cell and the elastic constants of the cell framework; it is greatest at high humidities. When all the fibrillæ are drawn into line, extension is retarded and afterwards takes place by direct extension of the fibrillæ. The parts of the cell structure which are not plastic in water at ordinary temperatures become so at higher temperatures, whilst at 100° extended fibres take a set which is permanent as regards subsequent immersion in cold water and is due to plastic flow within the fibrillæ. Different wools show fibrillar plasticity to different degrees, and for ease in manufacture wools should be highly plastic.

B. P. Ridge.

Some chemical aspects of wool research. A. T. King (J. Text. Inst., Spec. Issue, 1927, **18**, 361—368 T).—Clean wool removes hydrolysis-alkali from neutral soap solutions, the liberated fatty acids being partly deposited on the fibre as acid soap and partly left in

suspension in the liquid. In proportion to the amount of sodium hydroxide absorbed, the wool becomes yellowed by heating or steaming, acquires an increased affinity for dyestuffs, is more susceptible to bacterial attack, and less resistant to bleaching and chlorination. Wool keratin allows of no criterion of purity. Its sulphur content varies not only with variety, but also with fibres of different fineness from the same fleece. Approximately 66% of the total sulphur is removed in cystine, one of the most readily isolated of the hydrolysis products of wool, but although several products of amino-acid nature of known constitution have been obtained by the acid hydrolysis of keratin, no light has been shed on the nature of the keratin complex. In view of the changes in properties (*e.g.*, dyeing affinity) produced by exposure or chemical processing, it is suggested that some form of incipient hydrolysis occurs which, if it does not detach the simpler proteins, liberates their active groups by lactone or other ring formations. The anhydride of glycine (diketopiperazine) is suggested as representing the ring formation predominantly present in keratin, and its behaviour on hydrolysis as being similar to that of keratin ; a large number of substituted diketopiperazine ring formations are, however, possible. The cystine nucleus may act as an oxygen carrier in a similar manner to glutathione from which it is derived by hydrolysis, thus explaining the oxidation occurring on the exposure of neutral wool to light, and the behaviour of certain azo dyes on wool. B. P. RIDGE.

Isoelectric point of silk-fibroin. W. S. DENHAM and W. BRASH (J. Text. Inst., Spec. Issue, 1927, **18**, 520—524 T).—For samples of China and Italian silks, the results of experiments with copper acetate, silver nitrate, and potassium chromate place the isoelectric point between p_H 3·6 and p_H 4·0, the value 3·8 being accepted, although the possibility of the existence of more than one point, or of an isoelectric range, is not excluded. The p_H values were determined colorimetrically. No conspicuous difference is shown between the values for China and Italian silks or between those for mulberry and tussah fibroin. The isoelectric point for sericin of raw silk is about p_H 4. A considerable increase in the quantity of base fixed by fibroin from 0·02*N*-solutions of sodium or calcium chloride is shown when the p_H of the solutions is greater than 5, negligible amounts being absorbed near the isoelectric point.
B. P. RIDGE.

Nature of the action of sunlight on cotton. G. BARR and (Miss) I. H. HADFIELD (J. Text. Inst., Spec. Issue, 1927, **18**, 490—493 T).—Continuous decrease of strength and of the viscosity in cuprammonium solution, with increase in reducing power (as shown by the copper and silver numbers), are obtained with increasing time of exposure of cotton, whilst the Methylene Blue absorption shows first a fall and then a slow rise, the modification of cotton thus produced corresponding to that formed by mild oxidation with chlorine water. Experiments with linen show the reaction to be essentially one of oxidation, loss of strength occurring most rapidly in oxygen, less so in air, and to an almost negligible extent in a vacuum, hydrogen, or carbon dioxide. No significant change takes place when strips of cotton cloth are exposed for six months in sealed glass tubes containing hydrogen, either dry or about two thirds saturated with water vapour. Similar exposure in tubes containing oxygen results in considerable loss of strength with increase of copper number and the production of carbon dioxide and monoxide, and perhaps hydrogen, the reaction being more rapid in the presence of moisture and a rough parallelism being shown between copper number and the corresponding quantities of carbon dioxide produced. Similar cloth previously steeped in 0·001*N*-sulphuric acid is more highly reducing and shows a greater production of carbon dioxide after exposure under the same conditions. Precipitation of ferric hydroxide on the cotton before exposure (0·1% Fe_2O_3 on the weight of cloth) results in increased attack but diminished formation of carbon dioxide. It is suggested that at least two reactions may occur during the exposure of cotton to sunlight, both being accompanied by the development of reducing power and loss of strength, but one producing much less carbon dioxide than the other, iron being a photocatalyst to the latter reaction. B. P. RIDGE.

Ultra-violet radiation as an aid to textile analyses. H. R. HIRST (J. Text Inst., Spec. Issue, 1927, **18**, 369—375 T).—By the examination of substances submitted to the radiation from a mercury vapour lamp two types may be qualitatively distinguished : those showing pure brilliant colours and those having only ordinarily coloured appearances. Patterns of dyed fabric which have faded through exposure to light show considerable change in appearance under the influence of this radiation, and the method of examination furnishes a means of controlling the dyeing process in the production of shades fast to light. Many benzene substitution products, and azo dyes having benzene nuclei, show no fluorescence; similar naphthalene compounds and dyes are strongly fluorescent. α-Naphthol in aqueous alkaline solution gives a brilliant blue, β-naphthol a violet fluorescence, whilst salts of fluorescent compounds are more fluorescent than the acids or bases themselves. Solutions of fluorescent compounds (*e.g.*, α- or β-naphthol, or quinine sulphate) may be used as indicators for the titration of coloured or turbid solutions, since definite changes in the colour of the fluorescence are given at the neutral point. Textile fibres, and the same fibres in different stages of bleaching, give characteristic colours and can be thus identified ; the method also furnishes information about oils and oil, mildew, and other stains on wool fabrics. Unsaturated fatty acids give blue or violet, the saturated compounds white, and oxidised fatty acids yellow-brown or no fluorescence. Saturated hydrocarbon oils are non-fluorescent and unsaturated strongly so, although oxidation appears to destroy the source of fluorescence in oils. B. P. RIDGE.

Extensibility of flax yarns. J. A. MATTHEW (J. Text. Inst., Spec. Issue, 1927, **18**, 506—514 T).—A brief survey of published work with deductions therefrom. It is shown that stretch-load diagrams from flax yarns may be interpreted to reveal effects due to differences in the nature of the fibre in the yarns, and a measure is deduced which reveals the effects of past tensions and is an indication of irregularities in manufacturing conditions. B. P. RIDGE.

Some problems of textile testing. F. T. PEIRCE (J. Text. Inst., Spec. Issue, 1927, **18**, 475—489 T).—The influences of variable factors in the testing of textile materials are discussed from a mathematical point of view. B. P. RIDGE.

Determination of the physical properties of artificial silk and their relationship to textile manufacture. A. L. WYKES (J. Text. Inst., Spec. Issue, 1927, **18**, 494—505 T).—An artificial silk thread acts as though it consisted of two components, one brittle and one ductile, difficulties in weaving and knitting being ascribed to this dual nature. Its percentage extension under load increases very slowly with increasing tension until a yield point is reached, when it increases rapidly. Tension–% extension curves show that artificial silk can be converted into such a form that the thread is very brittle, very strong, and almost completely elastic, and it is suggested that the material under ordinary conditions contains a varying proportion of this brittle form of cellulose. For a thread tested first in a dry and then in a wet state the following relation holds: E/T for dry viscose $= E/1\cdot9T$ for wet viscose (where E is the % extension and T the tension), and from it the physical properties of any viscose silk (*e.g.*, yield point, elasticity, etc.) may be calculated. B. P. RIDGE.

Identification of cuprammonium rayon. ANON. (Textile Col., 1927, **49**, 242—243).—Copper (0·001—0·002%) in cuprammonium silks may be detected by ashing 3—5 g. in platinum, dissolving the ash in hydrochloric acid, adding excess of ammonia solution, and concentrating the filtrate to 5 c.c. One drop of potassium ethyl xanthate solution (5%) gives a yellow colour. Alternatively, a few drops of potassium ferrocyanide solution (5%) gives a pink coloration. With a concentrated solution, free from bromine, the addition of hydrobromic acid yields the red compound $CuBr_2,HBr,2H_2O$. CHEMICAL ABSTRACTS.

Structure of ramie cellulose as derived from X-ray data. O. L. SPONSLER and W. H. DORE (Fourth Colloid Symposium Monograph, 1926, 174—202).—The cellobiose (1 : 4) linking of the dextrose units does not exist in ramie fibre. Cellulose must consist of at least eight glucose units. The cellulose structure is stabilised longitudinally by the primary valency forces uniting the dextrose units, and laterally by the secondary valency forces of the oxygen atoms. Ester formation may alter the fibrous structure only by separating the longitudinal chains, but with the substitution of large groups the secondary valency forces may fail to stabilise the fibrous arrangement. CHEMICAL ABSTRACTS.

Microscopical structure of papermaking fibres in relationship to their manufacturing properties. J. STRACHAN (Proc. Tech. Sect. Papermakers' Assoc., 1925, **6**, 128—137).—From a study of microscopical structure it is possible to predict the behaviour of a fibre during beating and the character of the paper it will give. Thus, for example, thick-walled robust fibres hydrate slowly compared with hair-like fibres, which offer a greater effective area of adsorption. Factors determining strength include fibre length and thickness of cell wall, *i.e.*, individual fibre strength. Fibre characteristics associated with opacity are air-space within the fibre, a striated surface, and a laminated cell wall; those associated with closeness of texture (when no special beater treatment is to be given) are fineness of fibre and the presence of parenchymatous tissue. D. J. NORMAN.

Mechanism of resin sizing [of paper]. J. M. ARNOT (Proc. Tech. Sect. Papermakers' Assoc., 1925, **6**, 95—105).—The various theories put forward to explain the precise nature of resin sizing are reviewed and discussed. The author considers it possible that if, as suggested by Schwalbe, sizing is due to resin particles coated with aluminium hydroxide, the action of heat under suitable conditions causes the resin particles to coalesce to give a continuous film while still retaining a protective coating of alumina. Some support for this theory is adduced from the observation that sized fibres, when mounted in glycerin and immediately examined microscopically, expand slowly, whereas the resinous coating retains its original dimensions, later detaching itself and aggregating in coarse lumps, which appear to lie loosely among the fibres. Comparative laboratory experiments (using 4% of resin on the weight of pulp) show that resin sizing causes a considerable loss of strength (46·2% in the experiments cited). D. J. NORMAN.

Dyestuffs on cellulose acetate. KEYWORTH.—See IV.

PATENTS.

Degumming of silk. L. WALLERSTEIN. Assr. to WALLERSTEIN CO., INC. (U.S.P. 1,644,764, 11.10.27. Appl., 15.4.22. Renewed, 24.2.26).—Silk is subjected to the action of a proteolytic enzyme which is active in a faintly alkaline, neutral, or faintly acid medium at a temperature not less than 50°. D. J. NORMAN.

Preparation of cellulose for the manufacture of artificial silk etc. W. KAUFMANN, Assee. of M. KOHLER (E.P. 258,836, 5.7.26. Fr., 28.9.25).—Cellulose substantially free from hemicelluloses and suitable for the manufacture of artificial silk is produced from waste woven fabric by digesting the comminuted material, previously freed from foreign matter, for about 1—10 hrs. at 1—5 atm. with, *e.g.*, seven times its weight of a 3—20% suspension of caustic lime; digestion is preferably carried out in absence of air to prevent formation of oxycellulose. The digested material is washed, lightly bleached, again washed, and finally dried either in the form of flakes or in sheet form on drying cylinders. The resulting cellulose may be directly converted into alkali-cellulose by kneading it with twice its weight of 15—18% caustic soda solution. D. J. NORMAN.

Manufacture of artificial threads, filaments, etc. COURTAULDS, LTD., and F. D. LEWIS (E.P. 278,814, 19.7.26).—Improved results are obtained in the dry-spinning process if the temperature of the spinning solution and that of the air around the spinning nozzle are the same. This may be effected by, for example, passing the spinning solution through a coiled pipe placed in the upper part of the spinning chamber, or by using the same water to heat both the spinning chamber and the spinning solution. This method of dry-spinning enables the lustre and, to some extent, the cross-section of the

filaments to be accurately controlled when other conditions are constant; *e.g.*, when spinning a 20% solution of cellulose acetate in acetone (the air current being such that the concentration of acetone in the vicinity of the nozzle is about 1·5% by vol.) the lustre of the filaments decreases as the temperature falls from 67° to 62°, whilst a further drop in the spinning temperature considerably modifies the original round cross-section of the filaments. D. J. Norman.

Manufacture of artificial threads etc. and apparatus therefor. Courtaulds, Ltd., and C. F. Topham (E.P. 278,881, 11.10.26).—In the manufacture of threads of fine denier by the dry-spinning process difficulty is experienced when commencing spinning owing to the spinning solution spreading over the face of the jet instead of giving filaments. This may be avoided by applying to the solution passing to the jet a sudden pressure in addition to, and considerably in excess of, that supplied by the pump, for which purpose an auxiliary pump of the plunger type or a flexible diaphragm may be used. D. J. Norman.

Production of threads from viscose. C. A. Huttinger, Assr. to Acme Rayon Corp. (U.S.P. 1,646,538, 25.10.27. Appl., 14.8.22).—A soft, open thread is spun from the filaments obtained by introducing streams of viscose into a precipitating bath containing, by wt., 10 pts. of water, 6 pts. of sodium hydrogen sulphate, and 1½ pts. of sugar. H. Holmes.

Treatment of cellulosic materials. W. Kershaw, F. L. Barrett, and Bleachers' Assoc., Ltd. (E.P. 277,722, 23.4.26).—Cellulose which gives solutions (cuprammonium, viscose, and ester solutions) of low viscosity is obtained from cellulosic material by digesting the latter with 3—5% caustic alkali solution for, *e.g.*, 10—15 hrs. at 40—80 lb./sq. in., subjecting it to the action of a hypochlorite bleaching, and then, after washing, redigesting it with (1—5%) caustic alkali solution for about 5—15 hrs. at 10—80 lb./sq. in. A final bleaching operation may be carried out if desired. D. J. Norman.

Production of high α-cellulose [wood pulp]. A. E. White. From Brown Co. (E.P. 278,767, 13.4.26). —A continuous process for the production of wood pulp of high α-cellulose content from chemical pulp is described. Sulphite pulp, preferably produced with a sodium bisulphite cooking liquor containing 1% and 5—6% of combined and free sulphur dioxide, respectively, may be used with or without a preliminary treatment with a bleaching solution. The pulp passes through the apparatus as a suspension, efficient mixing at the various stages being ensured by reducing the consistency of the suspension to about 2%; pulp thickeners are used to increase the consistency again where necessary, *e.g.*, prior to digestion proper. Digestion is effected at 80—100° with 5—8% of caustic soda on the weight of bone-dry sulphite pulp at a consistency of 10—12%. The pulp is then washed, thickened to 10—16%, and bleached with 4—7% of bleaching powder (on the dry weight of pulp) at a temperature not above 32°. A further treatment—"super-bleaching"—may be given with a solution containing 0·2—0·5% Cl on the dry weight of pulp. After finally washing, the purified pulp is passed to a classifier where short or broken fibres (1—20%) are removed. The resulting product contains 94—96% of α-cellulose and 1—2% of pentosans, and has a copper number of 1·0—2·3. D. J. Norman.

Production of paper pulp. W. H. Howell, jun. (U.S.P. 1,645,754, 18.10.27. Appl., 10.9.26).—The digestion liquor is circulated by means of steam, circulation being maintained by letting off steam from the digester. The steam thus removed is used to heat the first effect of an evaporator through which the spent liquor is subsequently passed. D. J. Norman.

Bleaching of paper pulp. O. Kress, Assr. to Amer. Lakes Paper Co. (U.S.P. 1,645,061, 11.10.27. Appl., 21.8.24).—Sulphate pulp is treated first with a bleaching agent containing chlorine, and then with a sulphite. D. J. Norman.

Saccharification of wood. Internat. Sugar & Alcohol Co., Ltd. From Soc. Ind. de la Cellulose (E.P. 278,450, 9.7.26).—A more rapid and economical saccharification of wood is effected by treating sawdust (100 pts.) with less than the normal quantities of highly concentrated hydrochloric acid (40%) and gaseous hydrogen chloride, *e.g.*, 40—60 pts. and 15 pts., respectively, and then, after some hours, adding hydrochloric acid of medium concentration (above 35%, *e.g.*, 38%) to complete the saccharification and at the same time separate the sugar from the solid residue. D. J. Norman.

Pulping machines for paper mills. P. J. Wolff & Söhne, G.m.b.H., and H. Mallickh (E.P. 272,966, 21.6.27. Ger., 21.6.26).

Machine for the manufacture of paper [in continuous sheet]. W. H. Millspaugh (E.P. 278,817, 20.7.26).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Standardisation of the fastness of dyestuffs. S. G. Barker (J. Text. Inst., Spec. Issue, 1927, 18, 313—348 T).—A survey of the requirements in the establishment of standards of fastness to light. The violet carbon arc is suggested as the most suitable source of light in fading tests. Fading comprises simple loss of colour, colour change to a different hue, and loss of brightness, but not of colour, *i.e.*, the material looks dull. Its extent for exposures under natural conditions depends on the constitution of the sunlight and atmospheric conditions, humidity being of special importance; for testing, 75% R.H. is suggested as a standard. Fading is related to the wave-length of the incident light, and for experiments with a fadeometer the relation, $T = aL^n$, where T is the time of exposure in hours, L the percentage loss of the predominant colour, a is a numerical constant depending on the dyestuff, and n is a number of value about 2, holds approximately except for short exposures. For different depths of shade of the same colour the actual loss of colour for exposure under the same conditions is approximately constant. As increase in the ultra-violet radiation produces increase of fading, an artificial fading lamp should transmit only such radiation in the ultra-violet region as would be determined by the limits of the solar spectrum, if results

are to be compared with those due to sunlight. Several instruments, such as the tintometer, and the Eastman and Guild colorimeters, useful for purposes of colour matching, are described in detail. B. P. RIDGE.

Dyestuffs on cellulose acetate. KEYWORTH.—See IV.

Ultra-violet radiation [and dyeing of textiles]. HIRST.—See V.

PATENTS.

Dyeing of cellulose acetate silk. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 262,830, 14.12.26. Ger., 14.12.25).—In the application of ice-colours to acetate silk, carbamide or a carbamide derivative increases the avidity of the fibre for the dye, especially when added to the padding solution. When hydroxynaphthoic arylamides are used, formaldehyde may be added to the dye-bath (as with cotton). Examples are: 4-chloro-*o*-toluidine → 2 : 3-hydroxynaphthoic 2 : 5-dimethoxyanilide (red) or β-naphthylamide (red); *p*-nitroaniline → β-naphthol (yellowish-red). C. HOLLINS.

Dyeing of rubber (E.P. 241,214).—See XIV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

[Catalytic] oxidation of ammonia. G. B. TAYLOR (Ind. Eng. Chem., 1927, **19**, 1250—1252).—No upper limit of temperature for maximum yield in the reaction $4NH_3 + 5O_2 = 4NO + 6H_2O$ has been found. The practical limit is 1000° owing to wear on the platinum gauze. The rate of oxidation of nitric oxide is given by $-d[NO]/dt = k[NO]^2[O_2]$, and k increases 10% for each 10° drop in temperature. In the reaction between nitrogen peroxide and water at a given temperature and concentration of the liquid phase, $k_1 = [NO]/[NO_2]^3$, k_1 decreases rapidly as the concentration of liquid nitric acid increases; hence the difficulty of making directly nitric acid over 60% concentration. In some plants the first tower delivers weaker nitric acid than the second owing to the presence of excessive nitric oxide in the entering gases. With artificial refrigeration of the condensing system, the temperature being maintained at 0°, the effective capacity is doubled and the strength of the acid made increased from 50% to 60%. C. IRWIN.

Transformation of sodium chromate into dichromate by means of carbon dioxide. N. F. YUSHKEVICH and I. N. SHOKIN (J. Chem. Ind. [Russia], 1927, **4**, 204—207).—A process for converting sodium chromate into dichromate entirely with carbon dioxide is based on the observation that from concentrated aqueous solutions containing both salts, when heated at the b.p. (about 140°), sodium chromate is completely precipitated, whilst the dichromate remains dissolved. In practice, the sodium chromate solution (15 g. Cr per 100 c.c.) is heated in an autoclave at 5 atm. with lime-kiln gas (35 vol.-% CO_2) at 20° (or 10°) whereby 65% (or 75%) of the chromate is converted into dichromate. Above 3 atm. the sodium hydrogen carbonate is precipitated, and is removed in a centrifuge. The solution is then evaporated in a vertical vacuum apparatus (the separating chromate being collected) until it contains 62—69% of sodium dichromate, 9—5% of sodium chromate, and 29—26% of water. Evaporation (to 82% of dichromate at 145°) is completed in an open pan. The chromate separates completely, and the liquid is solidified in drums. CHEMICAL ABSTRACTS.

Precipitation of gold and silver from cyanide solution on charcoal. J. GROSS and J. W. SCOTT (U.S. Bur. Mines Tech. Paper, 1927, No. 378, 78 pp.).—Heat-treatment affects the capacity of charcoal (limits, 2000 oz. of gold, or 1000 oz. of silver per ton) but origin, quenching, or pulverisation finer than 200-mesh has little effect. After removal of the adsorbed salts with hot water, hot cyanide solution, or sodium hydroxide solution, the charcoal is used again. A unimolecular layer of cyanide appears to be adsorbed on the charcoal. CHEMICAL ABSTRACTS.

Synthesis of bentonites. M. NEKRICH (Ukraine Chem. J., 1926, **2**, 155—164).—By the interaction of solutions of aluminium sulphate and sodium silicate, substances are produced which are similar in composition and behaviour to the naturally occurring bentonites. Their general formula is given by $Al_2O_3,7SiO_2,nH_2O$, and microscopical examination shows a crystalline structure. They were found to be just as effective as the natural bentonites for adsorption of gases and purification of oils, but after regeneration by washing the waste products with ether and subsequent filtration, they were not so efficacious as the freshly prepared substances. Similar artificial bentonites obtained by substituting ferric for aluminium salts were not such good adsorbents or purifiers. A. RATCLIFFE.

Determination of small quantities of carbon monoxide in air. N. KOMAR (Ukraine Chem. J., 1926, **2**, 165—178).—Froboese's method (cf. B., 1915, 577), hitherto used only for small quantities of carbon monoxide, has been found very satisfactory for determining minute amounts (up to 0·01% by vol.) of the gas in air. A. RATCLIFFE.

Explosion risks in the use of liquid chlorine for bleach liquor. H. KIRMREUTHER and L. PURRMANN (Papier-Fabr., 1927, **25**, 698; cf. Schönberg, B., 1927, 875).—Schönberg's suggestion that an explosion which occurred in the preparation of bleach liquor with liquid chlorine was due to separation and explosive decomposition of solid calcium hypochlorite is unlikely both on chemical and on thermochemical grounds. The reaction between gaseous chlorine and milk of lime takes place with evolution of 290·7 kg.-cal. per kg. of chlorine, whilst the heat of vaporisation of liquid chlorine at 8° is only 62·7 kg.-cal./kg.; hence, provided that the lime is agitated and the temperature kept uniform, super-cooling and consequent separation of solid hypochlorite cannot occur. W. J. POWELL.

Spectrophotometric detection of boron. W. C. HOLMES (J. Assoc. Off. Agric. Chem., 1927, **10**, 522—523).—1 pt. of boron in 25 million pts. of aqueous solution may be detected by the increased absorption in the blue region of the spectrum on conversion of turmeric into the turmeric–boron compound. 1 c.c. of the aqueous solution acidified with 1 c.c. of glacial acetic

acid is evaporated to dryness with 2 c.c. of an alcoholic turmeric solution containing 50—60 mg. per 100 c.c., and the residue is dissolved in 5 c.c. of glacial acetic acid. A blank test using distilled water instead of the aqueous solution is carried out in the same manner. The extinction coefficients of the final acetic acid solutions are then determined in 2 cm. layers over the spectral range between 495 and 510 mμ. If boron is present in the solution under examination, the extinction coefficients obtained will exceed those of the blank.

F. R. Ennos.

Lead arsenate and lime spray mixtures. Van der Meulen and Van Leeuwen. **Isume phosphorite.** Roshdestvensky. — See XVI. **Determination of nitrogen.** Shedd.—See XIX.

Patents.

Production of hydrogen and phosphoric acid. E. Urbain (E.P. 278,578, 24.2.27. Fr., 7.12.26).—Hydrogen and phosphoric acid are produced from phosphorus vapours and steam in the presence of amorphous carbon at 350°, instead of at 600—1000°, by the use of hydrochloric, hydrobromic, or hydriodic acid as a catalyst. W. G. Carey.

Production of concentrated nitric acid free from oxides of nitrogen. Elektrizitätswerk Lonza (Gampel u. Basel), and E. Lüscher (Swiss P. 118,714, 20.11.25).—Aqueous nitric acid is treated with nitrogen peroxide and ozonised oxygen. L. A. Coles.

Regeneration of caustic alkali solutions. W. H. Allen (U.S.P. 1,647,764, 1.11.27. Appl., 25.2.27).—Alkali solutions used in the treatment of metal articles coated with paint, enamels, etc. are mixed with calcium hydroxide, and the precipitate is removed from the mixture. H. Royal-Dawson.

Apparatus for the purification of impure solutions of caustic soda etc. on osmotic principles. L. Cerini (E.P. 268,385, 28.3.27. Italy, 26.3.26. Addn. to E.P. 265,126; B., 1927, 329).—Diaphragms for use in the process of the prior patent are made of fabrics consisting of cotton or other vegetable fibre treated, without tension, with a caustic soda solution (*d* 1·36) at 40° for 24 hrs. H. Royal-Dawson.

Production of anhydrous chlorides. J. Gohin (Addn. No. 31,594, 5.11.25, to F.P. 601,612; B., 1926, 538).—Reaction and expansion chambers, which permit conversion of the chlorides into oxides, are provided at the end of the apparatus described previously.

L. A. Coles.

Manufacture of cyanides. N. V. Nederlandsche Mijnbouw en Handelmaatschappij (E.P. 262,802, 9.12.26. Ger., 10.12.25).—Material containing humic acid, *e.g.*, coal humus, and sodium carbonate are mixed, either in the semi-solid state or in solution if filtration is necessary, and, after the removal of water, the mixture is heated in a rotary oven at about 550° to form a compact, highly porous, alkali coke which is then heated at about 1000° in a current of nitrogen. Suitable catalysts, *e.g.*, iron compounds, may be added to the initial substance. W. G. Carey.

Production of sodium cyanide from calcium cyanamide. Deutsche Gold- u. Silber-Scheideanstalt vorm. Rössler (G.P. 442,966, 5.4.21).—A solution of the product obtained by fusing calcium cyanamide with sodium chloride by the usual process is concentrated until saturated with sodium cyanide, lime being precipitated before or during the concentration. L. A. Coles.

Separation of salts from solutions containing them. Siemens-Schuckert Werke G.m.b.H. (F.P. 619,828, 5.8.26. Ger., 27.8.25).—The solutions are atomised and cooled in a current of air, which then passes into electrical gas-purifying apparatus.

L. A. Coles.

Production of methane and carbon dioxide from carbon monoxide and hydrogen. A. Riégert (F.P. 619,621, 9.12.25).—The gas mixtures are subjected to high temperature and pressure in a bomb in the presence of a catalyst. L. A. Coles.

Manufacture of barium sulphide. G. Richardson (U.S.P. 1,648,870, 8.11.27. Appl., 8.4.25).—See E.P. 255,167; B., 1926, 788.

Separation of mixtures of gases (E.P. 273,229).—See I. **Purification of zinc solutions** (E.P. 278,851).—See X. **Cell for electrolysis of water** (E.P. 278,447).—See XI. **Barium and calcium sulphates** (E.P. 268,779).—See XIII.

VIII.—GLASS; CERAMICS.

Determination of the temperatures of the commencement of fusion and of tempering of industrial glass. E. Damour and A. Thuret (Compt. rend., 1927, **185**, 939—941).—The temperature at which fusion commences is denoted by an increase in the dilatation of the glass. The tempering and annealing temperatures of a number of industrial glasses have been shown to be equal, the former being determined by examining the effects of polarised light on rapidly cooled test pieces, and the latter from the point of separation of the two dilatation curves of the same glass tempered or annealed. The fusion and tempering temperatures may be confused with those of rapid and slow annealing, respectively. J. Grant.

Modern methods of testing refractories and their importance in gas-retort construction. E. Steinhoff (Gas- u. Wasserfach, 1927, **70**, 989—993, 1019—1023).—Whereas in high-temperature metallurgical furnaces the refractory materials suffer the greatest damage from dust and slag, for vertical gas retorts, which work at lower temperatures, the properties of the refractories requiring most consideration are linear expansion, constancy of volume, resistance to pressure at high temperatures, and thermal conductivity. A method of determining expansion is described, in which the sample is heated in a vertical furnace and the linear expansion determined to 0·001 mm. by measuring the angle through which a mirror, actuated by the expanding material, is deflected. The expansion of silica brick is much greater than that of firebrick, and its expansion–temperature curve shows a marked discontinuity at about 220°, corresponding to the cristobalite transformation. Alterations in volume are determined by heating a cylindrical sample in a muffle and measuring the volume before and after, by mercury displacement.

The temperature of initial softening of refractories depends on their previous history, and a more useful characteristic is the temperature at which the height of the sample is reduced by 30—40% under a load of 1 kg./cm.^{2} The order of increasing resistance is acid firebrick, basic firebrick, and silica. Determinations of thermal conductivity below 1000°, published by different investigators, show marked discrepancies, but above 1000° silica is definitely a better conductor than firebrick. Some indication of the homogeneity of a specimen may be obtained by etching with 10% hydrofluoric acid and staining with an organic dye. Carbon monoxide, in contact with heated refractories containing a trace of iron deposits carbon, which has a disruptive action on the brick, the effect being greatest at 450—500°. Methane behaves in a similar manner, most markedly at 900°. In silica and firebrick cements the particle size is of importance, also their behaviour under pressure at high temperatures. The linear expansion of silica cement is much greater before it has been heated to redness than after several hours' heating, but firebrick cement shows little difference due to heating.

W. T. K. BRAUNHOLTZ.

PATENTS.

Manufacture of refractories [for gas retorts etc.]. SOUTH METROPOLITAN GAS CO., and R. H. B. LAMPREY (E.P. 278,821, 23.7.26).—The plasticity and refractoriness of clay used in a refractory of clay and silica are increased by elutriation and acid washing, and the silica, consisting of tridymite and quartzite crushed to pass No. 4—No. 12 mesh, is added in such proportion that constant volume is maintained owing to the mutually compensating expansion of the silica and contraction of the clay.

W. G. CAREY.

Tunnel kilns (U.S.P. 1,646,208 and 1,646,279).—See I.

IX.—BUILDING MATERIALS.

Combination of lime in Portland cement compounds. W. C. HANSEN and R. H. BOGUE (Ind. Eng. Chem., 1927, **19**, 1260—1264).—The effect of the minor constituents of Portland cement on the completeness of the combination of the lime and on the temperature necessary for burning was studied by means of a tubular resistance furnace capable of control to 10°. The charge was contained in platinum boats which were moved forward every 2 min., thus imitating the conditions of the rotary kiln. For a cement containing 67% CaO, 10% Al_2O_3, 23% SiO_2, a maximum temperature of 1500° was required for complete combination of the lime. Replacement of small proportions of lime by soda or potash scarcely altered the temperature required, but replacement of lime by magnesia or alumina by ferric oxide markedly increased the combined lime or reduced the temperature of complete combination. With 3% MgO and 3% Fe_2O_3 the temperature of complete combination was 1325°. The principal effect of the alkali metals was an increase in fusion.

C. IRWIN.

Manufacture of artificial stone from blast-furnace slag. R. SCHÖNHÖFER (Arch. Eisenhüttenw., 1927, **1**, 267—271; Stahl u. Eisen, 1927, **47**, 1909—1910).—Slag from furnaces melting cast iron and from blast furnaces making hæmatite pig iron, and sufficiently basic slag from steel furnaces, may be utilised in the manufacture of artificial stone by grinding them to a fine powder in edge-runner mills with just sufficient water to produce a plastic mass. During grinding, ammonia and hydrogen sulphide are evolved and hydrolysis of some of the constituents of the slag takes place, so that the resulting mass is endowed with setting and hardening properties like cement. The mass is mixed with a proportion of sandy slag and with 2·5% of cement, and the mixture is pressed into blocks, which are set aside to harden for 8—10 weeks. Very basic slags are preferably mixed with a certain quantity of acid slag to reduce the basicity during the grinding operation.

A. R. POWELL.

Wood preservation. IV. Preservative properties of chlorinated coal tar derivatives. L. P. CURTIN and M. T. BOGERT (Ind. Eng. Chem., 1927, **19**, 1231—1234; cf. B., 1927, 909).—In order to find a cheap, highly toxic product capable of dissolution in petroleum, the toxicities of chlorinated coal-tar distillates were investigated. Creosote readily absorbs 20% of chlorine, but the product is little more toxic than untreated creosote. Naphthalene and neutral distillate (b.p. 240—270°) were less toxic after chlorination. Cresols and xylenols, however, showed a great increase in toxicity, dichloroxylenol being 100—150 times as toxic as creosote. High-boiling tar acids showed a reduction in toxicity after chlorination, due probably to the reduction in solubility. Such products as dichloroxylenol are stable in the atmosphere and have a low enough vapour pressure to be permanent in practice. They dissolve readily in petroleum distillate or acid-washed petroleum, but tend to form gum with petroleum residues. In the above, toxicity refers throughout to the fungus *Fomes annosus*.

C. IRWIN.

Protection of marine piling against borer attack. W. D. RAMAGE and J. S. BURD (Ind. Eng. Chem., 1927, **19**, 1234—1240).—A number of test pieces of wood treated with different distillate fractions of creosote oil were immersed for 3—4 years in sea-water. The protection against borer attack was in all cases good and no variation between the fractions was observed. On extracting the oils after 2½ years' immersion, however, it was found that practically all constituents of b.p. below 235° had been leached out. The percentage of such constituents therefore might be limited to the amount necessary for good penetration. Comparative tests showed that the rate of creosote loss from wood in sea-water was less than that in air, and this again less than the evaporation of creosote alone in an open dish. Extraction of the pieces with benzene, which entirely removes the creosote, showed that here again the loss was in the low-boiling constituents. A considerable proportion of the tar acids was lost, especially in water. There was no evidence of polymerisation or "fixation" by the wood. Investigations of the protective action of insoluble compounds of copper, mercury, and selenium precipitated in the wood by various methods resulted in failure in all cases. Tests with such concentrations of chlorine as it is possible to maintain in bay-water around piling showed that it is impracticable to kill *Teredo* by this means.

C. IRWIN.

PATENTS.

Preparation of organic materials or aggregates for use with cements. J. R. GARROW (E.P. 278,788, 7.7.26).—Organic material, such as wood shavings or sawdust, is expanded to above its maximum expansion under severe service conditions by impregnation with a hot solution of a salt (*e.g.*, lead chloride) which deposits insoluble crystals in the pores on cooling, or which, by further treatment with an appropriate reagent, combines with the reagent to form a solid filler or support to stabilise the material in its expanded condition. Mercuric chloride, calcium chloride, etc. may be used to act in addition as a preservative. W. G. CAREY.

Preparation of a metallic coating for walls, ceilings, etc. S. DE LANGE (E.P. 278,405, 4.6.26).

Impregnation of wood. J. R. COOLIDGE, Assr. to MONTAN, INC. (U.S.P. 1,648,294—5, 8.11.27. Appl., 26.1.26).—See E.P. 265,206; B., 1927, 909.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Potential measurements of non-rusting steels. H. STÄGER and H. ZSCHOKKE (Z. angew. Chem., 1927, **40**, 1265—1270).—Potential measurements of twelve chromium steels containing 0·09—0·47% C and 12·6—27·5% C, both after the usual heat-treatment and after air-hardening at 825—1100°, show that such steels invariably have a negative potential of about — 0·3 volt against the hydrogen electrode. After anodic polarisation, however, the potential becomes positive, varying from + 0·4 to + 0·5 volt, according to the composition. The hardening temperature has little effect on the oxygen potential except between 850° and 950°, when a well-marked minimum occurs corresponding with the minimum observed in the elongation–hardening temperature curve. Both the oxygen and hydrogen potentials of chromium steels become more negative with time of contact with the electrolyte, and the hydrogen potential becomes less negative after prolonged exposure of the metal to the air. These results support the oxide film theory of passivity. A. R. POWELL.

Potentiometric determination of the hardening elements in special steels. I. Determination of chromium and vanadium in the presence of iron. E. ZINTL and P. ZAIMIS (Z. angew. Chem., 1927, **40**, 1286—1291).—Addition of chromous sulphate, formed by dissolving chromous acetate in dilute sulphuric acid, to a solution containing chromate, vanadate, and ferric salts reduces first the chromate and vanadate to chromic and vanadyl salt, respectively, then the ferric salt to ferrous, and finally the vanadyl salt to vanadous salt. These stages may be determined potentiometrically, provided the iron present does not greatly exceed the sum of the vanadium and chromium, as follows. The slightly acid solution is boiled to expel air, cooled, treated with 1—2 c.c. of phosphoric acid (*d* 1·7), and titrated to the first potential jump. The solution is heated to 80°, treated with hydrochloric acid to give a 1—5% solution of that acid, and again titrated with chromous sulphate until the second and third potential jumps occur. In the presence of excess iron, good results are obtained only for the first stage; the following method is recommended in this case. The solution of chromate, vanadate, and ferric sulphate in 20% sulphuric acid is titrated cold with sodium arsenite to obtain the chromium and then with ferrous sulphate for the vanadium, the end-point in each case being determined potentiometrically. Manganese interferes as it is oxidised to the ter- or quadri-valent stage during the titration of the chromate, the arsenic acid formed acting as a stabiliser of the higher manganese compounds. A. R. POWELL.

Determination of slag and oxides in wrought iron. B. B. WESCOTT, F. E. ECKERT, and H. E. EINERT (Ind. Eng. Chem., 1927, **19**, 1285—1288).—The structure and properties of wrought iron are largely dependent on the presence of included slag. Eggertz's iodine method for the determination of slag content has been studied and the following modification is proposed: 3 g. of drillings are weighed into a 100 c.c. beaker, and 20 g. of resublimed iodine and 30 c.c. of distilled water are added. After the violence of the reaction has subsided, the contents of the beaker are stirred for 1—2 hrs. and filtered, and the residue is washed free from iron with boiling water. It is then treated on the filter with boiling 10% potassium hydroxide solution and again washed. The residue is dried and weighed. Silica and manganese may be subsequently determined. If the phosphorus content exceeds 0·15% some loss occurs owing to dissolution in the caustic potash. On the other hand, omission of the potash treatment may give high results if silicon exceeds 0·15%. The manganese in the slag is determined by oxidising the filtrate from the silica determination with sodium bismuthate and titrating with sodium arsenite. C. IRWIN.

Rapid method for the determination of sulphur in iron ores. C. C. HAWES (Min. and Met., 1927, **8**, 462—464).—The finely-powdered ore (0·5 g.) is intimately mixed with 2·5 g. of iron powder and the mixture is heated to bright redness in a nickel crucible in an electric furnace for 15 min., whereby all the sulphur is converted into ferrous sulphide, which is subsequently decomposed by treatment with hydrochloric acid (*d* 1·1). The hydrogen sulphide evolved is collected in 10% sodium hydroxide solution and determined iodometrically as usual. A. R. POWELL.

Structure of copper–zinc alloys. O. BAUER and M. HANSEN (Z. Metallk., 1927, **19**, 423—434).—The system copper–zinc has been reinvestigated by the thermal, micrographic, and electrical resistance methods, and various modifications of the accepted equilibria in the system are put forward. The positions of the lines AB and AD showing the beginning and ending of the solidification of the α-phase, the peritectic line BCD at 905°, and the α-(α + β) boundary BE agree with those found by Genders and Bailey (B., 1925, 246). The line CF separating the (α + β) from the β field extends from 63% Cu at 905° to 55·3% Cu at 453°, and is then vertical at 54·3% Cu down to the ordinary temperature. The $\beta \rightarrow \beta'$ transformation takes place at 453° in (α + β)-alloys and at 470° in (β + γ)-alloys; in β-alloys the temperature of the change rises with increasing zinc content from 453° to 470°. No evidence of two transformations as found by Jitsuka (A., 1927, 1141)

could be obtained, but the β-transformation is shown to take place 10° lower on cooling than on heating. The solubility of copper is about 1% greater in β than in β′. The solidus CG and the liquidus DH in the β range agree with those found by earlier authors except that the horizontal GH is at 833°. The boundary GJ between the β and (β + γ) fields joins the points 43·6% Cu, 833°, and 50·4% Cu, 470°, in a smooth curve, then falls to the axis of composition in a vertical slightly curved line, whilst the γ-(β + γ) boundary HK is convex to the temperature axis between 39·5% Cu at 833° and 42·3% Cu at 470°, and slightly concave to that axis between the latter point and 41% Cu at 20°. The peritectic LMN (γ + liquid ⇌ δ) is at 695°, the three points corresponding with 30, 27, and 19·5% Cu, respectively; the peritectic OPQ (δ + liquid ⇌ ε) at 594°, the three points corresponding with 23·5, 21·5, and 11·5% Cu, respectively, and the peritectic UVW (ε + liquid ⇌ η) at 423°, the three points corresponding with 12·5, 2·0, and 1·5% Cu, respectively. The dystectic reaction δ ⇌ (γ + ε) takes place at 555° along the line joining R (29·5% Cu), S (26% Cu), and T (21·5% Cu). The line separating γ from γ + (γ + ε) joins R to a point at 32·2% Cu at 400° in a smooth curve convex to the temperature axis and is then vertical to the composition axis. The field of pure δ is a triangle bounded by straight lines joining MOS, that of (γ + δ) a quadrilateral bounded by lines joining LMSR, and that of (δ + ε) a quadrilateral bounded by lines joining OPTS. Jitsuka's observation that there is no true eutectic at the zinc end is confirmed. A. R. POWELL.

Determination of copper [cuprous] oxide and metallic copper in mixtures containing both. W. D. BONNER and B. D. KAURA (Ind. Eng. Chem., 1927, **19**, 1288—1289).—Copper free from oxygen dissolves in alkali cyanide solution only to the extent of 0·02% in 3 hrs. at room temperature, in which cuprous oxide is dissolved to saturation. To 0·5 g. of the sample 50 c.c. of semi-metal sodium cyanide solution is added and the mixture kept for 1½—2 hrs. with occasional shaking. It is then filtered, treated with nitric acid, evaporated, redissolved, and freed from iron, and the copper content determined. Metallic copper is similarly determined from the undissolved portion. The method is suitable for the analysis of cement copper and of cuprous oxide. C. IRWIN.

Thermal decomposition of covellite and pyrite. A. C. HALFERDAHL (Min. and Met., 1927, **8**, 468—469).—From data obtained experimentally by Allen and Lombard (B., 1917, 454) and by Wasjuchnova (Diss., Berlin, 1909) the heats of decomposition of covellite and of pyrite to give solid solutions of sulphur in cuprous and ferrous sulphides, respectively, are —6600 g.-cal. (400—500°) and —10,000 g.-cal. (575—680°). A. R. POWELL.

Influence of chemical and crystallographic properties of cast metal on its behaviour during rolling. E. SEIDL (Min. and Met., 1927, **8**, 454—460).—Skillets and wire-bars of aluminium, zinc, and copper all have very similar structures in the cast state, characterised by large stalk-like crystals perpendicular to the cooling surface of the mould. If the skillets are rolled so that these crystals are perpendicular to the direction of rolling there is a tendency, most marked in the case of zinc, for intercrystalline cracks to develop between these fibrous crystals. This tendency is increased by the presence of impurities, such as lead and iron in zinc, $FeAl_3$ in aluminium, and cuprous oxide in copper, which segregate between the individual crystals, as well as by the occlusion of gases by the metal, which causes the formation of blowholes also between the fibres. Copper which is rolled parallel to the axis of the fibrous crystals often splits open along the middle, due to the action of the elongated blowholes between the thin laminæ produced on rolling the fibrous crystals. Good sheet may be obtained by rolling zinc bars which have been cast in a mould—slightly agitated until the metal solidifies—so as to produce a fine-grained crystal structure of no definite orientation. In the cases of copper and aluminium, however, such a structure can be obtained only after hammering and annealing (cf. Seidl and Schiebold, B., 1927, 77). A. R. POWELL.

Measurement of the oxidisability of aluminium and its industrial alloys after activation by mercuric chloride. C. QUILLARD (Compt. rend., 1927, **185**, 953—955).—A calorimetric method is described for the determination of the oxidisability of activated aluminium and its alloys by air in terms of the maximum rise in temperature as shown by time-temperature curves, the amounts of alumina formed being proportional to the rise. The rise decreases and increases with increasing proportions of manganese and magnesium in the alloy, respectively, whilst the presence of silicon (2—5%) has little influence. The maximum temperature is attained in about the same time for all the alloys. J. GRANT.

Influence of the casting conditions on the tensile properties of aluminium and magnesium alloys. G. SCHREIBER (Z. Metallk., 1927, **19**, 456—458).—The tensile strength and ductility of a large number of castings of the same alloy vary within certain limits. By noting the frequency with which every value recurs in a series of similar castings and plotting the figures so obtained against the corresponding tensile strength and ductility, curves are obtained which indicate the most probable tensile strength and ductility for any casting. Such curves have been obtained for cast iron, steel, electron, and some aluminium alloys. A. R. POWELL.

Analytical chemistry of tantalum, niobium, and their mineral associates. IX. Separation of titanium from tantalum and niobium. W. R. SCHOELLER and E. C. DEERING (Analyst, 1927, **52**, 625—637).—From a critical review and reinvestigation of more recent methods for the separation of titanium from tantalum and niobium, it is concluded that Hauser's mannitol method and those of Weiss and of Landecker are unsatisfactory. Muller's method (cf. B., 1911, 1114) was found to give the best results, not, however, as favourable as Muller reported. Titania cannot be extracted from a pyrosulphate-hydrolysis precipitate by salicylic acid or ammonium salicylate, but titanic acid precipitated by ammonia is soluble in either reagent. Fusion of the mixed oxides with potassium carbonate and extraction with water is quite ineffective; volu-

metric methods for oxidimetric determination of titanium and niobium in presence of tantalum are uncertain. In the authors' proposed separation method, the mixed oxides (0·1 —0·2 g.) are fused with 2 g. of potassium bisulphate, and the cold mass is dissolved by digestion with a strong solution of 3 g. of tartaric acid. After dilution to 300 c.c. the solution is boiled with 30 c.c. of strong nitric acid for 15—20 min., the precipitate (P^1) mixed with filter fibre, washed in 1% nitric acid, ignited, and weighed. If the weight is 0·1 g. or more, the procedure is repeated; the precipitate (P^2) is the earth-acid fraction. The combined filtrates from P^1 and P^2 are evaporated with 10—15 c.c. of strong sulphuric acid, and the tartaric acid is destroyed by cautious addition of nitric acid. After cooling and diluting, a slight excess of nitric acid is added, the precipitate mixed with filter pulp, washed with ammonium nitrate solution, ignited, and weighed as the titania fraction. The method, whilst giving very encouraging results, is still approximate. D. G. Hewer.

Zirconium. J. H. de Boer (Ind. Eng. Chem., 1927, **19**, 1256—1259).—Zirconium tetrachloride was prepared by heating the ore with carbon in a stream of chlorine at 800°. It was purified by melting with phosphorus pentachloride at 200—300° and distilling the mixture of double chlorides. The zirconium oxide was prepared by igniting the oxychloride prepared as above, and the crude metal prepared by reduction with calcium or sodium or by reduction of zirconium tetrachloride with sodium. The pure metal was prepared by treating the crude metal with iodine *in vacuo*, and allowing the tetraiodide to be reduced with deposition of the metal on a heated tungsten wire. The metal obtained was ductile, did not rust or tarnish, had m.p. 1850°, *d* 6·53, and tensile strength 95 kg./mm.^{2} The metal burns in air at a high temperature, and forms compounds at a red heat with chlorine, carbon, silicon, etc. If aluminium is present in the final purification an alloy is formed containing a compound of the two metals. C. Irwin.

Degasification of metals and its relation to corrosion. F. M. Dorsey (Ind. Eng. Chem., 1927, **19**, 1219—1225).—The difficulty of plating a metal with a really adherent coat of another metal which may be of any thickness, the plated metal being capable of being worked, is attributed to the presence of adsorbed or occluded gases beneath the plate. Degasification may be carried out by using the iron or steel to be plated as anode in a cell containing 96% sulphuric acid after the usual preliminary cleaning. A 12-volt direct current at 50 amp./sq. ft. is recommended. The original current decreases rapidly and approaches zero at the end of 3 min. The acid is then thoroughly removed with water and the object put quickly into the plating bath. Any base metal except aluminium can be treated successfully in this way, and plating with zinc, lead, copper, nickel, chromium, or vitreous enamel carried out. A soft nickel coat is produced in a bath containing nickel sulphate and chloride, boric acid, sufficient nickelous hydroxide to give p_H 4, and hydrogen peroxide. Bromothymol Blue is used as indicator and sodium must be absent. The temperature should be maintained at 55°. The bath is at first treated with chlorine to remove organic compounds, and the condition is maintained by additions of hydrogen peroxide. Iron and copper are also harmful. The current should be uniform (40 amp./sq. ft.). The acidity is kept constant by moving the nickel anodes as required. The bath should be of stoneware and perfect cleanliness maintained. Sections show that in plating produced in this way interlocking of the crystals occurs with no distinct boundary. Specimens may be rolled, pressed into shapes, or formed into tubes without injury to the plating. C. Irwin.

Improving [the corrosion-resistance of] metal surfaces by diffusion. G. Grube (Z. Metallk., 1927, **19**, 438—447).—The rate of diffusion of aluminium, tungsten, and molybdenum in iron and of chromium in nickel has been determined at different temperatures, and the results are reproduced in tabular and graphical form. No diffusion of aluminium takes place from an aluminium-iron alloy into electrolytic iron below the transformation point at 906°, but at higher temperatures diffusion is rapid; *e.g.*, at 1100° the surface layers of the iron contain 5—10% Al after a few hours. The rate of diffusion is rapid until the fibrous outer layer of iron has been penetrated, but slackens considerably when the regularly oriented crystal conglomerate in the interior is reached. Tungsten and molybdenum both diffuse rapidly into iron at 1300°, and the depth of penetration is relatively great. Chromium diffuses into nickel at 1200° somewhat more slowly and to a smaller extent than does aluminium into iron. In all the cases examined the graph connecting the content of the metal diffusing with the depth of penetration is a smooth curve up to a certain point, which varies with the metals used, the time of the test, and the temperature, after which there is a sudden abnormal drop in the curve indicating the presence of some factor which tends to restrain further penetration. The resistance of the treated surfaces to corrosion by nitric acid does not appear to follow Tammann's $n/8$ law in the case of chromium and nickel, but there is some correspondence with the rule in the case of chromium or aluminium diffusing into iron, in that there are sudden falls in the rate of dissolution when the molecular ratio Cr : Fe in the surface layers rises above 1 : 8 and again when it rises above 2 : 8, and when the Al : Fe ratio rises above 2 : 8 and 3 : 8, although these changes do not take place exactly at the right composition; possibly this deviation is due to uneven diffusion. A. R. Powell.

Aluminium ovens. Fischer and Tropsch.—See I. **Gold and silver from cyanide solution.** Gross and Scott.—See VII. **Stone from blast-furnace slag.** Schönhöfer.—See IX.

Patents.

Manufacture of sponge iron from its oxide ores. W. W. Percy, Assr. to F. W. Harris (U.S.P. 1,645,968, 18.10.27. Appl., 13.3.22).—A zone of a descending column of ore is subjected to the action of hot gases produced by combustion of hydrocarbon fuel outside the column. Hydrocarbon fuel is introduced into the column above the zone, and gases withdrawn from the upper part of the column are regenerated and returned to the column below the zone to complete the reduction of the ore. H. Holmes.

Reduction of iron ores. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 278,167, 29.3.26).—Reducing gases at a temperature of 800—1300°, prepared from powdered fuel and oxygen or oxygenated air, are used. The oxygen or oxygenated air may be mixed with water vapour, carbon dioxide, or gases already used in the reduction, and the reducing gases may be produced either in a separate generator or in contact with the molten iron by blowing the powdered fuel mixed with oxygen on to or into the melt. The reduced iron may be melted by bringing the hot gases into contact with it before employing them for reduction. The used gases, and particularly the carbon monoxide contained in them, may be treated with steam and then employed for other chemical purposes. M. E. Nottage.

Manufacture of metals and alloys [*e.g.*, ferromanganese] of low carbon content. G. E. R. Nilson (E.P. 260,232, 5.10.26. Swed., 22.10.25).—A manganese ferrous silicate slag of high manganese content is smelted with carbon to obtain ferromanganese with a high carbon content, which is then melted in an electric arc furnace with quartz and a slight deficiency of carbon to produce a silicomanganese with a relatively low carbon content (0·5%) and more than 15% Si. This alloy is poured into a molten mixture of manganese ore and lime contained in a second arc furnace, whereby a vigorous reaction occurs with the formation of ferromanganese containing about 0·5% Si and 0·3% C and a slag containing about 25% Mn as manganous calcium silicate. This slag is then used in the first stage of the process. A. R. Powell.

Treatment of magnetic material. [Improving the magnetic properties of alloy steel sheets.] W. E. Ruder, Assr. to Gen. Electric Co. (U.S.P. 1,648,697, 8.11.27. Appl., 23.1.24).—Alloy steel sheets, after removal of a surface layer, are annealed under conditions which favour the removal of deleterious gases. J. S. G. Thomas.

Reduction of ores etc. W. Buddéus (E.P. 264,834, 18.1.27. Ger., 20.1.26).—A mixture of ore or the oxides of easily fusible heavy metals (*e.g.*, lead, tin, antimony, bismuth) and silicon or silicides is introduced into liquid slag from a blast furnace working the same metal, the thermic reaction being sufficient to effect a reduction and separation of the metal. The reaction agents are preheated sufficiently to ensure the necessary conserved heat for the scorification of the gangue and impurities. C. A. King.

Treatment of ores etc. containing titanium and iron. C. A. Klein and R. S. Brown (E.P. 279,219, 17.9.26).—The slag obtained by smelting titaniferous iron ores according to E.P. 243,081 (B., 1926, 99) is freed from suspended iron particles by granulation, crushing, and magnetic separation, or by treating the stream of molten slag with a jet of high-pressure steam and subjecting the disintegrated slag directly to magnetic separation. A. R. Powell.

Manufacture of articles from sintered hard-metal alloys. F. Krupp A.-G. (E.P. 278,955, 18.3.27. Ger., 25.10.26).—Tools etc. are made from a sintered hard-metal alloy, *e.g.*, tungsten carbide, which is free from uncombined carbon. Tungsten carbide for this purpose containing 5·2—6·2% C is formed by heating tungsten powder with carbon in the slightly oxidising atmosphere of a gas furnace, or by heating an exact mixture of the components in a current of hydrogen in an electric furnace. C. A. King.

Chlorination of ores. A. Ramén (E.P. 255,475, 15.7.26. Swed., 15.7.25).—The ore and chlorinating agent are intimately mixed, formed into briquettes or moulds, and then heated to such a temperature that the chlorinated metallic compounds are volatilised and can be collected in a suitable device, whilst the residue remains in the briquette form. The briquettes may be placed either on a travelling trough moving in a tunnel furnace and heated by any ordinary means, or on a movable grate, covered with a layer of combustible material, and the latter ignited, the air required for burning being introduced either by forcing it in below the grate or by withdrawing the burning bases from above it. M. E. Nottage.

Recovery of tin from tin-plated materials. J. Jacobsen, J. Goffin, L. Goffin, and L. Renson (E.P. 255,092, 9.7.26. Ger., 9.7.25).—The materials are treated with a hot solution of chlorine in hydrochloric acid, the chlorine being replaced continuously as the operation proceeds; the detinned metal is removed and washed. The tin is recovered from the solution by electrolysis, the electrolyte being prepared either by dividing the solution into two portions, one of which is made alkaline to precipitate tin hydroxide, which is then dissolved in the other portion and acidified with sulphuric acid; or the solution is concentrated and cooled, the crystals which separate are redissolved in dilute sulphuric acid, and the solution is brought to the right degree of concentration to form the electrolyte. M. E. Nottage.

Extraction of [platinum] metals from materials containing them. A. R. Powell, E. C. Deering, and Johnson, Matthey & Co., Ltd. (E.P. 279,156, 19.7.26).—Ores or residues containing the platinum metals in the metallic form or in a form easily converted into the metallic form (*e.g.*, by roasting and reduction) are ground in a tube mill with a dilute solution of sodium hydroxide or ammonia containing a soluble copper salt, *e.g.*, sodium copper cyanide, copper tartrate, or a cuprammine, and an amalgam of a metal, *e.g.*, zinc, which will replace copper from the solution. The platinum metals become coated with copper and are then taken up by the mercury, from which they may be recovered by any suitable process. A. R. Powell.

Treatment of zinc waste. E. Bury (E.P. 278,411, 24.3.27).—The solution obtained by dissolving the waste in sulphuric acid is treated with an excess of ammonia so as to form soluble double compounds of zinc and ammonium, impurities such as iron and aluminium hydroxides together with any gelatinous silica being removed by sedimentation and filtration. The filtrate is boiled down to precipitate zinc hydroxide, which is collected on a filter, whilst the remaining solution is evaporated to obtain ammonium sulphate crystals. The zinc hydroxide is subsequently calcined to oxide. M. E. Nottage.

Purification of zinc solutions preparatory to recovery of zinc by electrolysis. Rhodesia Broken Hill Development Co., Ltd. From R. H. Stevens, G. C. Norris, and W. N. Watson (E.P. 278,851, 1.9.26).—Impure zinc solutions (especially of zinc sulphate) which have been obtained from zinc ores etc. are purified for the electrolytic production of zinc by the addition of a xanthate or allied derivative of dithiocarbonic acid appreciably soluble in the zinc solution. Zinc xanthate is preferable, being added to the basic, neutral, or slightly acid solution. Copper, cadmium, cobalt, nickel, arsenic, antimony, and mercury are precipitated as insoluble xanthates. If 0·2—0·3 g. of copper per litre is not initially present, an addition of a soluble copper compound is made to facilitate precipitation. B. Fullman.

Electrolytic deposition of chromium. General Motors Corp., Assees. of W. M. Phillips and P. W. C. Strausser (E.P. 254,757, 6.7.26. U.S., 6.7.25).—Chromium is deposited upon a conducting surface from a solution containing 200 g. of chromic acid and less than 12·5 g. each of chromium sulphate and boric acid per litre. The temperature of the bath is maintained at 45—60° and a current density of 125—200 amp./sq. ft. is used. The rate of deposition of chromium may be increased by agitating the bath by the passage of air through it or by movement of the cathode. M. E. Nottage.

[Coating and] impregnating metals and other materials with aluminium or aluminium alloys. W. Smith (E.P. 279,273, 14.12.26).—Iron or steel articles are coated with cadmium by electrodeposition, then coated with aluminium by immersion in molten aluminium (or its alloys) or by exposure to aluminium vapours. In both cases the cadmium is removed completely from the article, by dissolution in the aluminium in the first case and by volatilisation in the second. A. R. Powell.

Heat-treatment of metallic pieces. M. Fourment (E.P. 263,774, 1.12.26. Fr., 29.12.25).—Metallic pieces, *e.g.*, wire, bars, strips, etc., are passed through a tube which may be evacuated or put under the pressure of a gas, and which is wound with an inductive coil forming part of a high-frequency electric circuit. J. S. G. Thomas.

Treatment of ores for extraction of values. F. Dietzsch (U.S.P. 1,648,760—1, 8.11.27. Appl., [a] 8.5.25. Belg., 25.10.23. [b] 10.8.25).—See E.P. 226,258; B., 1925, 104.

Manufacture of bearing material. C. Claus (U.S.P. 1,648,722, 8.11.27. Appl., 28.5.26).—See E.P. 275,444; B., 1927, 784.

Metallurgical furnace. B. Talbot (U.S.P. 1,648,247, 8.11.27. Appl., 16.3.27. U.K., 5.3.26).—See E.P. 273,779; B., 1927, 659.

Rotary furnaces (E.P. 268,308 and 278,774).—See I.

Zinc white from zinc (Austr. P. 105,794).—See XIII.

XI.—ELECTROTECHNICS.

Ultra-violet radiation in textile analysis. Hirst.—See V. **Potential measurements of steels.** Stäger and Zschokke, also Zintl and Zaimis. **Aluminium wires as conductors.** von Zeerleder and Bosshard. **Galvanising of duralumin.** Haas and Pöttken.—See X. **Scheele's Green.** Bruns.—See XIII. **Electrodialysis of soils.** Clark and others.—See XVI. **Water purification by electro-osmosis.** Behrman.—See XXIII.

Patents.

Induction furnace. J. M. Weed, Assr. to Gen. Electric Co. (U.S.P. 1,648,707, 8.11.27. Appl., 14.12.23).—A crucible forming a reservoir for the charge communicates with a looped chamber within which a portion of the charge, forming a closed secondary, is heated inductively by current flowing in a primary winding. Means are provided to cause the charge to flow spirally through the looped chamber and to return through the reservoir. J. S. G. Thomas.

Induction furnace. W. Hoskins (U.S.P. 1,646,862, 25.10.27. Appl., 3.6.27).—In an induction furnace comprising conducting material contained within a curved riffled trough supported in a furnace setting, means are provided for supplying material to and withdrawing material from the trough and for inducing an electric current in the conducting material so that it is set in rotation. J. S. G. Thomas.

Recuperative [electric] furnace. F. T. Cope, Assr. to Electric Furnace Co. (U.S.P. 1,647,725, 1.11.27. Appl., 21.12.25).—A recuperative hood communicates with each end of the furnace heating chamber, and means are provided for passing strips of material through the furnace in opposite directions and in contact with each other, so that heat is conducted directly from one strip to the other. J. S. G. Thomas.

Electric furnace. F. T. Cope and R. F. Benzinger, Assrs. to Electric Furnace Co. (U.S.P. 1,647,726, 1.11.27. Appl., 6.3.26).—Plates forming the furnace hearth are mounted upon piers, between which a resistor grid is supported between the hearth and furnace floor. J. S. G. Thomas.

Electric tunnel kiln. J. Kelleher, Assr. to Harper Electric Furnace Corp. (U.S.P. 1,647,524, 1.11.27. Appl., 26.6.25).—The current through an electric resistor heater placed in one part of the tunnel is controlled by means of a resistor heater rheostat placed in another part of the tunnel and adapted to control the temperature of that part. J. S. G. Thomas.

Gaseous-discharge lamp. C. C. Van Voorhis, Assr. to Westinghouse Lamp Co. (U.S.P. 1,647,591, 1.11.27. Appl., 21.6.23).—Alkali metal is distilled *in vacuo* and the distillate collected in a capillary tube, a portion of which, containing a desired amount of alkali metal, is detached and placed in a tubulure attached to the lamp bulb. The end of the tubulure is then sealed, and the tubulure heated so that alkali metal distils and condenses on the walls of the lamp bulb and is finally "tipped-off." J. S. G. Thomas.

Electric-discharge device. [Rectifier.] E. E. Charlton, Assr. to Gen. Electric Co. (U.S.P. 1,648,293, 8.11.27. Appl., 29.11.22. Renewed 23.9.27).—An electric rectifier, designed to operate at a temperature at which the vapour pressure of cæsium is about 1 micron of mercury, comprises a thermionic cathode and an anode

adapted to operate at a substantially lower temperature than the cathode, enclosed in an envelope filled with gas at a pressure of several cm. of mercury and containing a quantity of alkali metal of greater atomic weight than that of potassium. J. S. G. THOMAS.

Production of a homogeneous mixture for coating metallic conductors composed of [metals of] the platinum group with a view to improving their electronic emission. SÜDDEUTSCHE TELEFON-APPARATE-, KABEL-, & DRAHTWERKE A.-G. (G.P. 442,833, 28.2.23).—The finely-powdered active material is suspended in a hydrocarbon compound of high mol. wt., *e.g.*, colophony or paraffin; the suspension is allowed to settle and congeal and the top layer, amounting to 20—25% of the total mass, skimmed off.

J. S. G. THOMAS.

Electrolytic cell [for electrolysis of water]. A. E. KNOWLES (E.P. 278,447, 9.7.26).—The upper end of the cell is closed by a cover having one or more ventilating traps which permit air to pass freely into or out of the cell, and which are charged with an absorbent, *e.g.*, soda-lime, for extracting carbon dioxide from the air during its passage. J. S. G. THOMAS.

Electrolytic diaphragms composed of glass threads. J. BILLITER (G.P. 442,965, 24.6.23. Austr., 23.4.23).—Membranes composed of layers of long parallel threads of glass or quartz, preferably extending throughout the length of the diaphragm and enclosed in frames, are employed. Such diaphragms are employed, *e.g.*, in the electrolysis of alkali chlorides.

J. S. G. THOMAS.

Diaphragm for water electrolyser. BAMAG-MEGUIN A.-G. (Swiss P. 118,710, 28.12.25).—The upper part (10—20% of the total height) of the edge of a diaphragm consisting of a gauze of non-rusting alloy, *e.g.*, nickel-chromium steel, is very closely woven.

J. S. G. THOMAS.

Galvanic cell. A. HEIL (G.P. 443,010, 6.9.25).—The carbon electrode, used more especially in conjunction with chromic acid in a galvanic cell, is made non-porous by impregnation with paraffin etc., and is made very many times larger than the zinc electrode used with it. J. S. G. THOMAS.

Galvanic cell having an electrode consisting of manganese dioxide and acetylene soot. P. BURGER (E.P. 259,220, 30.9.26. Ger., 30.9.25).—The soot to be used is produced by the decomposition of a mixture of acetylene with other hydrocarbon vapours, *e.g.*, benzene, benzine, gas oil, etc. H. ROYAL-DAWSON.

[Insulation of the electrodes of] electric [smelting] furnaces. A.-G. BROWN, BOVERI, & CIE. (E.P. 272,227, 2.6.27. Ger., 7.6.26).

Drying of charged battery plates. SILICA GEL CORP., Assees. of E. W. HUTCHINSON and W. J. PLEWS (E.P. 255,059, 5.7.26. U.S., 13.7.25).

Apparatus for electro-osmosis (F.P. 619,080).—See I. **Carbon granules for microphones** (U.S.P. 1,646,389).—See II. **Separation of salt solutions** (F.P. 619,828).—See VII. **Magnetic properties of alloy steel** (U.S.P. 1,648,697). **Heat-treatment of metallic pieces** (E.P. 263,774).—See X.

XII.—FATS; OILS; WAXES.

Detection of hardened fats. F. WITTKA (Chem. Umschau, 1927, **34**, 295—296).—The methods of detecting hardened fats, based on the tests for (*a*) traces of nickel and (*b*) *iso*- or solid unsaturated acids, are criticised as cumbersome. It is observed that whereas beef tallow and its products normally contain 0·27% of unsaponifiable matter, and at most 0·35%, the animal and vegetable oils usually used for hardening purposes, as rape, sesame, linseed, soya, whale, seal, sardine, and herring oils, have much higher values varying between 0·75 and 1·40% (castor oil is an exception with a value of 0·2%). Since these figures remain substantially the same after the oils are hardened, the determination of unsaponifiable matter affords a direct test for the presence of hardened fats. E. HOLMES.

Detection of hardened fat in tallow by determination of the iodine values of the solid fatty acids separated by Twitchell's method. S. C. L. GERRITZEN and M. KAUFFMAN (Chem. Weekblad, 1927, **24**, 554—556).—The iodine value, determined by Winkler's method, of the fatty acids separated from beef fat by the Twitchell method, is always below 6; a higher iodine value indicates admixture, either with mutton fat, for which the corresponding value may be as high as 13, or with hardened vegetable oils, for which, owing to the presence of the so-called *iso*oleic acid and related acids, the corresponding value may be as high as 27. If there has been no admixture with mutton fat the percentage of hardened vegetable oil in a commercial beef fat may be calculated with fair accuracy from the iodine value of the separated fatty acids.

S. I. LEVY.

Grape seed oil industry. J. BONNET (Bull. Soc. d'Encour., 1927, **126**, 523—541; cf. Carrière and Campredon, B., 1927, 562).—A review of the industry describing the early efforts to utilise the grape seed as a source of oil, and the development of the process of solvent extraction, by means of trichloroethylene, of the oil in France. The main requirements for economical working of the process are: (*a*) factories situated in proximity to large available supplies of raw material; (*b*) storage of the seeds with free access of air to prevent mould formation; (*c*) sorting of the grape seeds from the adherent pulp containing 50—60% of moisture by an appropriate beater; (*d*) drying of the seeds by causing them to travel by means of a worm-propeller which is subjected to steam heat; (*e*) solvent extraction of the dried seeds in which not more than 4 kg. of solvent are lost per 100 kg. of oil produced, not more than 0·6—0·8% of oil being left in the seeds. For a factory to be commercially workable, 1,500,000 kg. of marc are required; this will yield 300,000 kg. of seed and 600,000 kg. of pulp cake, the seed yielding about 25,000 kg. of oil and a further 200,000 kg. of seed-cake. The seed cake contains 2—2·5% N, 0·15—0·25% P_2O_5, and 0·5—0·6% K_2O. H. M. LANGTON.

Physical and chemical characteristics of some grape seed oils from France and Algiers. E. ANDRÉ and H. CANAL (Bull. Soc. d'Encour., 1927, **126**, 542—559).—An examination of the oil from the seed of 46 different varieties of grapes from various parts of France

and Algiers is reported, the oil being extracted in the laboratory by means of light petroleum. The oil content of the seeds was 6·5—18·2%. The oil had d 0·912—0·9264; n_D 1·4738—1·4802; saponif. value, 176·1—189·6; acetyl value (André) 2·4—43·2; iodine value (Hanus) 125—157; acidity as oleic acid, 0·5—36·1%. The values obtained for six samples of oil obtained on the factory scale were: d 0·9094—0·9338; n_D 1·4732—1·4761; saponif. value 181·2—206·0; acetyl value (André) 27·6—72; iodine value (Hanus) 93·6—137·3; acidity as oleic acid 4·4—59·8%. The author favours the view that variability of properties is a feature of grape seed oils, and seeks to correlate this with the variability known to exist in the 21 species of vine (*Vitis vinifera*) which exist in upwards of 500 varieties. It is concluded that in respect of the oil, the erucic acid type exists, but its iodine value departs noticeably from that of oils of the *Cruciferæ*, and that the ricinoleic acid type also exists, but ought to be unusual and appears to be produced in vines growing in hot climates (*e.g.*, North Africa). Most of the grape seed oils are of the semi-drying type.

H. M. Langton.

Grape seed oil. L. Margaillan (Bull. Soc. d'Encour., 1927, **126**, 560—561).—The conflicting results and corresponding conclusions drawn by various investigators when determining the constants of the oil are criticised, and the contention that oil extracted from fresh grape seeds resembles castor oil whilst that from seeds in course of change more and more resembles linseed oil is rejected by the author. Unlike castor oil, grape seed oil is insoluble in alcohol, exhibits no striking viscosity, and has a low acetyl value except when the oil is extracted from seeds which have been damaged, but even then the acetyl number never reached 50. Exposure of the seed to inclement conditions or a long interval before the crushed seeds are extracted permits rapid oxidation of the oil, but there is no relationship between increase in acetyl value and acid value, which remains low. The acetyl value of the oil is not a constant but a variable, as is the case with most oils. The oil is compared with rape oil, though it is blown less easily than the latter. . Grape seed oil blown for 12 hrs. gave an acetyl value of 57 (set), whereas the value for rape oil blown for only 9 hrs. was 94·5. H. M. Langton.

Determination of free fatty acid of oil in seed. R. K. Brodie, C. H. Cox, and W. D. Hutchins (Oil and Fat Ind., 1927, **4**, 177—181, 190).—The seed (100 g.) is heated for 30—45 min. at 100—105°, cooled, the meats are separated and ground to pass a 1·5 mm. sieve. The oil is extracted from 10 g. by cold percolation with petroleum (b.p. $< 70°$), neutralised alcohol (30 c.c.) is added, and the acid titrated. Chemical Abstracts.

Simple determination of the oil content of oil seeds. G. Brackman (Masloboino scirovoie gelo, 1926, No. 9).—The seeds (4 g.) are mixed with ether (100 c.c. less the estimated oil volume), the extract is filtered (evaporation of the ether being avoided), and the residue left on evaporation of 50 c.c. is weighed. It is claimed that the method is not less accurate than the Soxhlet method. Chemical Abstracts.

Rancidification and oxidation of olive oil. L. L. Lloyd (J. Text. Inst., Spec. Issue, 1927, **18**, 517—519 T).—The rancidity of olive oil is due chiefly to the marc or foots, which either acts as a fermentative agent or aids bacterial action. Moisture plays only a small part in the production of fatty acids. By passing, respectively, dry and moist air through samples of (*a*) oil containing foots, (*b*) the same oil free from foots, and (*c*) a mixture of equal parts of both, for periods up to three years, it was shown that the fatty acids are almost completely oxidised, whereas the neutral oil is oxidised only to a small extent. The larger the content of fatty acids, the more is the neutral oil oxidised; it appears that the former have a catalytic oxidising effect on the latter. It is advantageous to filter fresh oil to free it from foots before storing. B. P. Ridge.

Action of α-naphthol as negative catalyst in oxidation of drying oils. R. S. Morrell (J. Oil and Colour Chem. Assoc., 1927, **10**, 278—289).—The addition of small amounts of α- (and to a lesser degree β-)naphthol delays the drying of a drying oil or oil-varnish and also opposes the tendency to "blooming," possibly by retarding the formation of superficial peroxides. The increases of weight of various oil films when oxidised in filtered air on glass plates in a desiccator are recorded and graphed, the reduction in rate of gain of weight caused by the introduction of α-naphthol being marked even when strong positive catalysts, *e.g.*, manganese or cobalt resinate, are present. The retardation of oxidation is also confirmed, using the Mackey oil tester. The inhibitor has no apparent effect on the surface properties of drying oils, the differences being manifest only in respect of oxidation changes. In the presence of cobalt driers, phenol, tricresyl phosphate, and heptalin (in contradistinction to α-naphthol), have no noticeable effect on the "blooming," drying, webbing, or skinning-over of tung oil and tung-oil varnishes. Previous work on this subject is recapitulated and the views of Moureu and Dufraisse on "anti-oxygènes" are corroborated. S. S. Woolf.

Oil of *Centrophorus granulosus*. A. C. Chapman (Analyst, 1927, **52**, 622—624).—Further work on the unsaturated hydrocarbon spinacene, obtained from a sample of "Barroso" oil (*Centrophorus granulosus*), indicates its probable identity with Tsujimoto's squalene, and shows that the products formed by decomposition of the ozonide were very similar to those recorded by Majima and Kubota, from squalene. After removal of the bulk of the spinacene, cholesterol and an alcohol (m.p. 69°) were isolated. The alcohol was found to have a composition agreeing with the Japanese batyl alcohol. The remaining liquid alcohol, contaminated with about 10% of spinacene, was hydrogenated and a product (m.p. 68—69°) obtained, which was selachyl alcohol. The amount of glycerol obtained corresponded with about 0·5—0·6% on the original oil. Further, an examination of the fatty acids showed that the liver oil contained, in addition to the unsaturated hydrocarbon, cholesterol, batyl and selachyl alcohols, glycerol, stearic, palmitic, oleic, and possibly smaller proportions of other saturated and unsaturated fatty acids. The impossibility in many cases of separating the hydrocarbon and alcoholic constituents of oils, *i.e.*, the so-called unsaponifiable matters, from the soaps produced by saponification of the esters is noted. D. G. Hewer.

Capybara oil. R. A. D. da Silva (Rev. brasil. med. pharm., 1926, 2, 313—317, 493—494).—The oil from *Hydrochoerus capibara*, L., has d^{15} 0·9124—0·9164, d^{25} 0·9112—0·9150, n^{25} 1·46788—1·46986, n^{40} 1·46128—1·46326, saponif. value 196·93—197·87, iodine value (Hübl) 98·55—107·95, (Hanus) 97·21—115·48, ester value 194·79—195·53, acid value 1·40—3·08. Neither cholesterol nor lipochrome was found. Vitamin-*A* could not be detected. Chemical Abstracts.

Determination of iodine value in aqueous solutions [of oils]. A. Fialkov (Ukraine Chem. J., 1926, 2, 139—142).—An emulsion of the oil is formed by grinding it with half the amount of gum arabic and two drops of water. A further 10 c.c. of water are then added, drop by drop at first, the emulsion is well mixed, and finally the mixture is transferred to a flask with a ground stopper. After the addition of 20 c.c. of 0·5*N*-solution of iodine in potassium iodide the solution is made up to 200—250 c.c., again mixed, and, after 5 min., titrated with 0·1*N*-sodium thiosulphate, using starch as indicator. A control experiment is performed at the same time. The iodine value so found for various oils, *e.g.*, castor, linseed, cod liver, and cotton-seed oils, agreed with values obtained by Hübl's method. A. Ratcliffe.

Separation point of rape oil and aniline. C. E. Klamer (Chem. Weekblad, 1927, 24, 556—557).—The temperature of separation of a mixture of 1 pt. of rape oil with 4 pts. of aniline is lowered by free fatty acids; the acids of soya bean oil have approximately the same effect as the acids of the rape oil itself. Lowering may be due also to oxidation products, so that account must also be taken of these, since oxidation may not always be responsible for rancidity.
S. I. Levy.

Fats from yeast manufacture. Bendecky.—See XVIII.

Patents.

Apparatus for the distillation of solvents in connexion with the extraction of oils, fats, waxes, etc. L. J. Simon, and Simon Bros. (Engineers), Ltd. (E.P. 278,815, 19.7.26).—The apparatus is intended for use in conjunction with E.P. 255,923 (B., 1927, 49), where, instead of being intermittent, continuous distillation of solvents from a mixture of oils and solvents is desired. The method consists in treating the mixture in a preliminary distiller of the usual type which itself is made to control, by means of a ball float, the quantity of solvent to be evaporated by a number of smaller stills arranged in series, parallel, or series-parallel, the whole to provide for a continuous distillation of the liquid under treatment. The small distiller vessels (stills) contain closed steam coils and the end still may contain both closed and open steam coils. Each small still contains a tray having a heating coil along which the solution travels and is provided with baffles for circulating liquid around the tray, heating coils in the body of the still, and adjustable means for controlling the depth at which liquid is removed from each still. The vaporising of solvent from a solution gradually lowers the level of solution and permits the flow of fresh solution to the small stills at a rate which increases as the solution in the preliminary distiller tank becomes more concentrated. H. M. Langton.

Extraction of oleaginous and other materials with volatile solvents. J. Savage (E.P. 278,891, 22.10.26).—In a process which is particularly applicable to materials containing substantial amounts of moisture, *e.g.*, the degreasing of bones, the extraction is performed at pressures in excess of atmospheric and the separated solvent is returned to the distillation zone at a temperature just below its b.p. at the existing pressure of the system. In the extractor apparatus is a float-valve separator contained within a separating chamber exposed to the working pressure whereby the condensed volatile solvent and water are separated from each other. The apparatus comprises a container divided into upper and lower chambers by a perforated partition, heating coils being in the lower and a condensing coil in the upper chamber; a tray is provided to receive condensed vapour, a sprinkler coil below the tray, and an external pipe conveying condensed vapour to a water-separating chamber with a float-actuated, water-release valve. A pipe returns the hot separated solvent to the lower chamber. The apparatus is not limited to volatile solvents heavier than water, but can be used with those lighter than water by a modification of the release valve. H. M. Langton.

Apparatus for the extraction of oils and fats [from fish etc.]. K. Shjler (Can. P. 264,956, 3.7.25).—The apparatus comprises toothed rollers working in conjunction with two superposed drums in such a manner that grinding of the material and expression of the oils and fats take place simultaneously. L. A. Coles.

Production of soap. A. Welter (E.P. 254,755, 5.7.26. Ger., 3.7.25).—To achieve the partial or complete substitution of the water in a hydrous soap by sodium bicarbonate, split or distilled fatty acids, with or without the addition of fat solvents, are intimately mixed with powdered calcined soda until a homogeneous soapy mass is obtained, the weight of soda being at least 30% of the fatty acids, but not exceeding double the amount required for complete saponification. Before or after saponification is completed, liquid or solid grain soap or soap paste is added in suitable proportions (dependent on the nature of the soaps) and the product subjected to a milling process. Ease of milling and reduction in the amount of fatty acid necessary are claimed *inter alia* (cf. E.P. 202,710; B., 1923, 1032 A).
S. S. Woolf.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Agreement of an accelerated and an exposure test of structural paint. E. D. Gregory (Ind. Eng. Chem., 1927, 19, 1225—1229).—The details and results of a 13 years' exposure test on various structural paints are described, photographs being given in typical cases. A marked increase in the life of the paint system is achieved by using a special red lead (80% of red lead: 20% of special clay of extreme fineness and individual qualities) in place of ordinary red lead in the priming coat. The advantages of substituting varnishes, treated oils, etc. for part of the vehicle of a straight linseed oil paint are also illustrated. The results are in virtually complete agreement with those of accelerated tests on the same paints carried out in 1913 by Dubois (B., 1914, 32). A *résumé* of this earlier work is given. S. S. Woolf.

Electrochemical preparation of Scheele's green. B. P. Bruns (Ukraine Chem. J., 1926, 2, 143—154).—When a 5—10% sodium sulphate solution saturated with arsenic trioxide was electrolysed using a copper anode a compound was obtained very similar to Scheele's green in colour and composition. The best product was obtained when electrolysis was carried out at 80°. Current density and deposition of cuprous oxide on the anode influence the yield. Schweinfurt green can be similarly obtained by adding sodium acetate to the electrolyte. A. Ratcliffe.

Technical varnish film investigation. J. D'Ans (Chem. Umschau, 1927, 34, 283—291; 296—304).—[With S. Merzbacher.] The general phenomenon of respiration during the hardening of films of drying oils is discussed, and a review of the literature of the subject, particularly in regard to the bearing of autoxidation, polymerisation, and colloidal reactions on the process, is presented. [With K. Weise.] Apparatus is described whereby the amounts of oxygen absorbed, and the gaseous products evolved by a linseed oil varnish, during periods varying between two days and three months, can be determined. The easily volatile products of the dried film are then examined by heating the linoxyn at 130° for 5 hrs. in a stream of nitrogen. In the experiments of longest duration the varnish absorbed 39% O, and evolved 10% CO_2 and 1% CO (approx.). The carbon dioxide evolution was accelerated by sunlight and an elevated temperature, but these factors had no influence on the rate of evolution of carbon monoxide. Of the products volatile at 130°, 12·3% was water, 10·4% soluble acids expressed as formic acid and of which 7·2% was identified as formic acid, 0·8% aldehydes expressed as formaldehyde, and 1·5% (approx.) carbon dioxide, all calculated on the weight of the applied varnish. Altogether 36% of products volatile at 130° were obtained from the reaction. The formic acid is considered to be a degradation product of more complex cleavage products of the process, and is not derived from glycerol, which remains in the free state in oxidised linseed oil varnish films. On the basis of these results a tentative oxygen balance of the reaction is arrived at, and it is deduced that 6½ atoms of oxygen are retained in the linoxyn for each mol. of triglyceride originally present in the oil. E. Holmes.

Oxidation of drying oils. Morrell.—See XII.

Jalap. Dale.—See XX.

Patents.

Addition of materials to paints to prevent separation and settling of pigments. R. Schlick (G.P. 442,650, 25.9.24).—A small percentage of a suitable material is intimately mixed with the vehicle of a paint before incorporation of the pigment, in order to prevent adsorption of resinous constituents on to the pigment particles with consequent settling out. S. S. Woolf.

Preparation of pigment-oil compositions. G. W. Acheson (E.P. 265,541, 23.7.26. U.S., 5.2.26).—Oil is added gradually and preferably continuously to a well-stirred, pasty, pigment-water-oil emulsion, in which the oil is the dispersed phase, at a rate not substantially exceeding that at which the oil can be absorbed. The nature of the emulsion is thereby preserved until a separation of water occurs; the resulting homogeneous and smooth pigment-oil paste is then suitably dried. [Statutory ref. to E.P. 27,312 of 1907.] S. S. Woolf.

Zinc white from metallic zinc. C. R. Beringer (Austr. P. 105,794, 14.5.24).—Zinc, volatilised by heat radiated from above (the walls and roof of the heating channel being electrically or otherwise suitably heated), is converted into zinc oxide in a current of air. Pure oxide may be obtained from raw material containing, *e.g.*, 60% Zn, the impurities sinking to the bottom of the chamber and being drawn off. S. S. Woolf.

Manufacture of pure white and finely-distributed barium and calcium sulphates. T. Lichtenberger and L. Kaiser (E.P. 268,779, 29.3.27. Ger., 3.4.26).—Crude heavy spar, gypsum, or anhydrite is powdered and dissolved in molten alkali chlorides or other suitable salts. After removal of insoluble sediment, lime is added and air or water vapour is blown into the molten mass, whereby further sedimentation of impurities occurs. The purified material is cooled rapidly by, *e.g.*, granulation in water, and further treated with water to extract soluble alkali chlorides etc. S. S. Woolf.

Manufacture of composite titanium pigments. C. A. Klein and R. S. Brown (E.P. 278,791, 8.7.26).—Barium carbonate is added to the boiling water into which a paste of barium and titanium sulphate is to be poured (cf. E.P. 243,081; B., 1926, 99). The sulphuric acid liberated on the hydrolysis of the titanium sulphate, hitherto waste, reacts with the carbonate. The addition of hydrochloric acid or other acids capable of yielding soluble barium salts which are decomposed by sulphuric acid is also claimed. S. S. Woolf.

Manufacture of lacquers etc. I. G. Farbenind. A.-G. (E.P. 262,818, 13.12.26. Ger., 14.12.25).—Lacquers are obtained by treating condensation products of carbamide or its derivatives and formaldehyde, preferably in an organic solvent, with an equal or greater amount of cellulose esters, resins, cyclic ketones, or other organic substances capable of forming solid solutions with the condensation product. S. S. Woolf.

Organic moulding composition. S. M. Hull, Assr. to Western Electric Co., Inc. (U.S.P. 1,648,719, 8.11.27. Appl., 13.6.24).—A protein is condensed with a substantially anhydrous aldehyde. J. S. G. Thomas.

Manufacture of [phenol-aldehyde] condensation product. M. Melamid (U.S.P. 1,648,858, 8.11.27. Appl., 6.5.20. Ger., 27.12.18).—See E.P. 137,291; B., 1921, 520 A.

Resin from gutta-percha (E.P. 278,922).—See XIV.

Solvent for use in varnishes (E.P. 275,652).—See XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Colloid chemistry of rubber latices. Determination of actual and potential alkalinity of latex from *Hevea brasiliensis*. E. A. Hauser and P. Scholz (Kautschuk, 1927, 304—305).—The electrometric method is unsatisfactory for the measurement of the p_H of

rubber latex, and methods based on the colour change with indicators, particularly Wulff's method employing a film stained with an indicator, are preferable. From the moment of tapping until shortly before coagulation the p_H of latex is between 7·2 and 7·0, and then falls to 6·9—6·6. The higher values obtained by earlier workers are attributed to experimental error. The limited range of variation in p_H is ascribed to the existence of an equilibrium between hydroxyl ions in the solution and those adsorbed at the surface of the rubber globules. Determination of the potential alkalinity by titration using bromothymol blue or phenolphthalein as indicator reveals more distinctly a decrease in the alkali content of the latex prior to spontaneous coagulation. D. F. TWISS.

***Hevea* latex. VII. Rubber derived from preserved latex.** R. O. BISHOP (Malayan Agric. J., 1927, **15**, 271—282).—Rubber from ammonia-preserved latex vulcanises more rapidly than rubber from fresh latex; the difference is even more marked with rubber coagulated with alcohol than with rubber coagulated with acetic acid. Investigation of the proportion of amide nitrogen and amino-acids in preserved latex reveals no increase commensurate with the alteration in the rate of vulcanisation. It is possible that the observed changes in vulcanising properties are associated with the presence in latex of a labile complex substance (cf. Belgrave, B., 1924, 265; 1925, 771), which is affected by variation in the hydrogen-ion concentration of the latex. D. F. TWISS.

Variation in plantation rubber. B. J. EATON and R. O. BISHOP (Malayan Agric. J., 1927, **15**, 283—289).—Examination of about fifty samples of rubber from various Malayan estates shows that although the rate of vulcanisation in a rubber 90 : sulphur 10 mixture varies very considerably, the tensile properties of the vulcanised product are notably uniform. Of twenty-four samples of smoked sheet rubber nineteen had an optimum period of vulcanisation between 3 and 4 hrs., the normal figure for smoked sheet being $2\frac{1}{2}$—$2\frac{3}{4}$ hrs. D. F. TWISS.

PATENTS.

Concentration of the globuloids in rubber latex. M. S. STUTCHBURY, Assee. of W. BACHMANN, F. HEBLER, and C. BÖHM VON BÖRNEGG (G.P. 442,856, 2.10.23).—The concentration of latex by filtration, centrifuging, or evaporation is facilitated by previous treatment with an agglutination agent, *e.g.*, a solution of an aluminium salt, with the previous addition, if desired, of substances to prevent coagulation. D. F. TWISS.

Dyeing of india-rubber etc. METZELER & Co. (E.P. 241,214, 9.10.25. Ger., 9.10.24).—India-rubber, gutta-percha, and similar substances in solution in organic media or in aqueous suspension are treated with solutions of suitable dyestuffs, *e.g.*, acid, vat, or sulphur dyes, which can subsequently be converted into an insoluble form. D. F. TWISS.

Manufacture of rubberised material and compositions for same. GEN. RUBBER Co., Assees. of M. C. TEAGUE (E.P. 250,167, 14.9.25. U.S., 1.4.25).—Materials such as concrete, wood, stone, cork, or various fibrous materials which are not easily wetted by water are treated with a sulphonated oil (or *o*-toluidine, sodium sulphanilate, sodium salicylate, thiourea, or ammonium linolenate or *iso*linolenate) and an artificial or natural aqueous dispersion of rubber. The former, which facilitates wetting, may be applied before or together with the rubber emulsion. A suitable proportion of the wetting agent is 5% calculated on the rubber. The rubber dispersion may contain compounding and other ingredients. D. F. TWISS.

Accelerating vulcanisation [of rubber]. SILESIA VER. CHEM. FABR. (E.P. 270,644, 18.2.27. Ger., 4.5.26).—Vulcanisation of rubber is accelerated by salts of substituted guanidines with inorganic acids, *e.g.*, by di-*o*-tolylguanidine thiosulphate. D. F. TWISS.

Manufacture of a solid resin from the semi-fluid resinous matter extracted from crude gutta-percha and/or balata. A. B. CRAVEN and YORKSHIRE DYEWARE & CHEMICAL CO., LTD. (E.P. 278,922, 1.1.27).—The semi-fluid resin obtained from gutta-percha and/or balata, *e.g.*, by extraction with cold petroleum spirit, is agitated and treated with a current of air in the presence of a siccative, *e.g.*, 1% of manganese resinate; during the oxidation process the temperature is raised from 60° to 100° by external heating. The resulting solid resin has a pale amber colour and usually contains approximately 3% of free acids. For the purpose of neutralising these 0·5% of quicklime may be added to the liquid resins before oxidation. D. F. TWISS.

Manufacture of finely-divided substances from natural [rubber] emulsions or suspensions. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ, and F. R. MOSER (E.P. 278,395, 7.4.26. Addn. to E.P. 245,418; B., 1927, 100).—The process of the prior patent is applied to the coagulation of natural emulsions (*e.g.*, rubber latex). The latex is coagulated in the presence of finely-divided, colloidal substances such as clay, aluminium hydroxide, silica, etc. which may be produced during the coagulation by suitable chemical reactions. The product obtained, "latex gel," is miscible with all materials used in the manufacture of india-rubber goods, with substances for the preparation of mineral aggregates, with paper pulp for the manufacture of waterproof paper, etc. The gel is much more stable than ordinary or preserved rubber latex. R. C. ODAMS.

Vulcanisation process and product. A. C. BURRAGE (E.P. 279,280, 4.1.27).—See Can. P. 245,929—30; B., 1926, 453.

XV.—LEATHER; GLUE.

Action of sodium sulphide solutions in the manufacture of sole leather. V. CASABURI (Ledertech. Rundschau, 1927, **19**, 105—111, 117—124, 141—146).—Experiments have been carried out with solutions (*d* 1·004, 1·007, 1·010, 1·014, and 1·018) of freshly-prepared sodium sulphide, commercial sodium sulphide (alone and with sodium chloride), ammonium chloride, and calcium chloride, respectively, on dried hides. The hydroxyl and hydrosulphide ions absorbed by the hides increased as the strength of the solutions increased, and in the latter case proportionally. The results obtained were better in every case than

with lime alone. A solution of sodium sulphide (d 1·0105) containing sodium chloride gave the best yield and quality of leather, and prepared the pelt for tanning. The use of sodium salts prevents the precipitation of the tannin by lime in the pelt. The function of deliming is not to remove the lime but to provide a thin, acid pelt layer between the alkaline pelt and the tannin, until the tannin and pelt have combined. This layer should always precede the penetration of the tannin. The acid is not for swelling but as an aid to tannage. The strength of the tan liquors is unimportant if a suitable amount of acid is present. The collagen fibres are not split up into fibrils by alkaline swelling, but the membrane encasing the collagen fibre becomes elastic and stretches to its maximum length as the swelling increases. Acid swelling splits up the collagen fibre into fibrils, consequently the leather produced is soft and fallen. The pelt should be in an alkaline swollen condition at the moment of the fixation of the tannin; there must therefore be an acid intermediary layer on both surfaces of the leather at p_H 5·0 (approx.). D. WOODROFFE.

Welted insole leather. H. BRADLEY and A. COLIN-RUSS (J. Soc. Leather Trades' Chem., 1927, 11, 329—336).—Different samples (51) of vegetable-tanned insole leathers were analysed for moisture, fat, water-soluble matter (official), free tannin, ashed water-soluble matter, ashed insoluble matter, fixed tannin, degree of tannage, density, and true water-soluble matter. The water-soluble matter per unit of hide substance was calculated from the results, and the leathers are divided into three groups: (a) hide substance >38%, (b) 38—30%, and (c) <30%. The higher the hide substance, the lower was the value of the water-soluble matter per unit of hide substance; $e.g.$, <0·5, 0·5 to 0·9, and >0·9, respectively, for the above hide substance groups. The other analytical figures for leathers in these respective groups show that the latter are groups of quality. Leathers which have caused staining of hose are to be found amongst those in the third (or possibly the second) group. Insole leathers should be analysed by the official methods and graded according to the content of hide substance. The values for ash, fat, and ratio of free tannin to water-soluble matter in the respective groups are: (a) 2·5%, 2·0%, 0·7; (b) 3·5%, 4·0%, 0·6; (c) 5·0%, 4·5%, 0·5. D. WOODROFFE.

Extraction of tannins and water-soluble [matter] in leather analysis. J. CHARTERS (J. Soc. Leather Trades' Chem., 1927, 11, 350—351).—The neck of an inverted, wide-mouthed bottle, from which the bottom has been cut off, is fitted with a cork through which passes a small-bore capillary tube bent back on itself to form a siphon, the latter being covered by a test tube to prevent clogging. A flask (1 litre or larger) is used as a reservoir above the bottle and is fitted with a two-hole cork, through which pass an air vent and a long glass tube bent twice at right angles, which is connected by a rubber tube and screw clip to another glass tube delivering water from the flask into the bottle. The leather to be extracted is placed in the inverted bottle, covered with water to below the siphon, kept overnight, and water allowed to run in drop by drop. The rate of flow and the temperature of the water are regulated, and the extraction is intermittent. D. WOODROFFE.

Utility and solvent action of different fat solvents in the determination of fat in leather. R. LAUFFMANN (Ledertech. Rundschau, 1927, 19, 63—65, 71—74).—The solvent action of fats and oils in leather is diminished by their formation of compounds with the hide substance or of calcium, magnesium, and chromium soaps. Certain non-fatty substances, $e.g.$, tannins, sulphur, are dissolved by fat solvents, their solubility being increased by the presence of moisture. Samples of leather were treated with weighed amounts of degras and Turkey-red oil, respectively, and the dry leather was extracted after 2 and 120 days with solvents such as carbon disulphide, chloroform, ethyl ether, light petroleum, and carbon tetrachloride. The amount of fat extract was less in every case than the calculated; the amounts of natural grease extracted varied from 1·6% with light petroleum to 2·4% with ether. Chloroform and carbon disulphide yielded the most extract. The author does not agree with Woodroffe's explanation of the increased amount of extract from damp leather (cf. B., 1924, 529). The low yield of extract from leather fat-liquored with Turkey-red oil is attributed to the soap present in it and to the partial insolubility of the sulphonated constituents. D. WOODROFFE.

PATENTS.

Tanning of shark skins. A. EHRENREICH (E.P. 278,885, 14.10.26).—Shark skins are tanned, the dermic dentelli are removed, and the skins are given a supplementary tanning or "super-tanning" by any known process. D. WOODROFFE.

Dissolution and reprecipitation of collagen or glutin. M. BERGMANN and H. KOESTER (G.P. 442,520, 1.2.25).—Collagen or glutin, or materials containing them, are extracted separately in a definite order or together with solutions of metal ammines, $e.g.$, cuprammonium hydroxide. The solutions may, if desired, be mixed with metal ammine solutions of other substances, of vegetable or animal origin. The final product is precipitated with acids or other substances capable of decomposing the metal ammine compound. Threads can be prepared by forcing the solutions of collagen and/or glutin through fine orifices into a sulphuric acid precipitation bath. D. WOODROFFE.

Production of swollen or liquid animal glue. W. VON RECHENBERG (G.P. 442,046, 29.10.25).—Carbonates, which neutralise acids formed by oxidation with liberation of carbon dioxide, are added to the glue in addition to the usual preservatives. L. A. COLES.

Solvent for use in shoe-creams (E.P. 275,652).—See XX.

XVI.—AGRICULTURE.

Nitrates and wheat yields after certain crops. P. E. KARRAKER (Soil Sci., 1927, 24, 247—258).—The percentages of nitrates in soil samples from plots cropped under different rotations have been determined. The experiments extended over 5 years, the samples being taken in the autumn when wheat was sown. The

figures for the last 3 years indicate a fairly close relation between nitrate production and wheat yields; over the whole period the relationship is only very general.
C. T. GIMINGHAM.

Effects of liming and green manuring on crop yields and on soil supplies of nitrogen and humus. C. A. MOOERS (Tenn. Agric. Exp. Sta. Bull., 1926, No. 135, 64 pp.).—The principal effect of liming, in rotation of cowpeas with wheat, was to increase the amount of available soil nitrogen. Such stimulation accords with the law of diminishing returns.
CHEMICAL ABSTRACTS.

Variable occurrence of nitrates in soils. P. E. KARRAKER (Soil Sci., 1927, **24**, 259—262).—Large variations in the amounts of nitrogen as nitrates in individual samples of soil from apparently uniform plots are reported.
C. T. GIMINGHAM.

Nitrogen losses through dentrification [in soils]. A. M. BUSWELL and S. L. NEAVE (Soil Sci., 1927, **24**, 285—290).—A summary of recent literature on the subject, with bibliography.
C. T. GIMINGHAM.

[Yield from] barley of different nitrogen content. ANON. (J. Dep. Lands Agric. Ireland, 1927, **26**, 333—334).—Apparently poor seed containing 1·8% N gave slightly better crops than apparently good seed (of the same stock) containing 1·25% N.
CHEMICAL ABSTRACTS.

Effect of hydrogen-ion concentration on absorption of phosphorus and potassium by wheat seedlings. J. DAVIDSON (J. Agric. Res., 1927, **35**, 335—346; cf. Davidson and Wherry, B., 1924, 484).—Wheat seedlings grown in potassium phosphate solutions absorbed relatively more potassium than phosphorus. In solutions with initial p_H 5·0 or lower, this preferential absorption of potassium resulted in increased acidity, and more phosphorus was absorbed from these solutions than from those of p_H 6·0 or 7·0. The physiological availability of phosphorus thus appears to depend on the p_H value of the medium. The excess of phosphorus absorbed from the acid solutions was found in the tops of the seedlings. The roots from the neutral solutions contained more phosphorus and almost twice as much potassium as those from corresponding acid solutions. Absorption phenomena of this type may be explained by the assumption that there is a relatively wide range in the isoelectric points of the individual protoplasmic ampholytes of the cells.
C. T. GIMINGHAM.

Reactions between monocalcium phosphate and soils. R. H. AUSTIN (Soil Sci., 1927, **24**, 263—269).—Titration of monocalcium phosphate with calcium oxide leads to the formation of tricalcium phosphate and calcium monohydrogen phosphate; with calcium carbonate the latter only is formed. The view that aluminium renders phosphorus highly insoluble in an acid soil medium is not confirmed, since little aluminium phosphate is formed when monocalcium phosphate is titrated with aluminium hydroxide. In general, a change from acid to alkaline reaction is accompanied by a rapid decrease in the phosphorus in solution; nevertheless the soluble phosphorus may be extremely low in some very acid soils, indicating that some factors other than soil reaction or the content of iron and aluminium determine its amount.
C. T. GIMINGHAM.

Production of available phosphates from Isume phosphorite. I. G. ROSHDESTVENSKI (Ukraine Chem. J., 1926, **2**, 179—194).—In order to convert the phosphoric acid present in Isume phosphorite into a form easily assimilable by a method other than conversion into a superphosphate, the material was treated with portions of a solution of 1% sulphuric acid and *N*-hydrochloric acid. It was found that lime was extracted each time, first in smaller, then in larger quantities; phosphoric acid was similarly extracted, the amount present in each extraction depending on the concentrations of sulphuric acid. Further replacement of the hydrochloric acid by various salts (*e.g.*, sodium, potassium, or magnesium chlorides) showed that the amount of lime extracted depended on the nature of the salt used in the solvent. Vegetational experiments indicated that treatment of the phosphorite with a mixture containing 0·5*N*-sulphuric acid, 0·15*N*-sodium chloride, and 0·1*N*-magnesium chloride gave the best increase (36·6%) in the crop of barley.
A. RATCLIFFE.

Mutual effects between plant growth and the change of reaction of the nutrient solution with ammonium salts as the source of nitrogen. T. L. LOO (Japan. J. Bot., 1927, **3**, 163—203).—Knop's solution was modified by the use of sodium nitrate and inorganic ammonium salts instead of calcium nitrate as the source of nitrogen. In contact with the roots, the solution containing ammonium nitrate, chloride, or sulphate becomes more acid, that of ammonium hydrogen phosphate or carbonate changes little, whilst that containing sodium nitrate becomes less acid except for buckwheat, horse-bean, and lupins. The inferiority of ammonium salts as the source of nitrogen for higher plants is ascribed to the increase in hydrogen-ion concentration of the solution during growth; addition of sodium phosphate or calcium chloride to ammonium chloride or sulphate is beneficent, as it retards the rise of acidity.
CHEMICAL ABSTRACTS.

Buffer capacity of soils and its relation to the development of soil acidity from the use of ammonium sulphate. W. H. PIERRE (J. Amer. Soc. Agron., 1927, **19**, 332—351).—A collodion-sac method of obtaining the soil extract is described. In greenhouse experiments (supported by field experiments) the use of 500 lb. of ammonium sulphate per acre caused the acidity of two of fourteen soils to exceed p_H 4·8; 1000 lb. per acre caused six soils to reach this or a lower value.
CHEMICAL ABSTRACTS.

Relativity of the terms "alkalinity" and "acidity" as applied to soils. J. CLARENS (Bull. Soc. chim., 1927, [iv], **41**, 1383—1387; cf. B., 1926, 762).—In the presence of other salts, *e.g.*, sodium, calcium, or ammonium chlorides, the results obtained for the alkalinity of soils by the method previously described are modified considerably owing to adsorption of salts by the soil or to replacement of some of the soil base by the metal in solution. Sodium salts generally make the soil appear to be more alkaline, and calcium and ammonium salts more acid than it really is.
A. R. POWELL.

Percentage of carbon dioxide in soil air. C. O. Appleman (Soil Sci., 1927, **24**, 241—245).—Analyses of the soil air in rows of potatoes in the presence of different amounts of organic matter are recorded. Incorporation of organic matter increased the percentage of carbon dioxide in the soil, the increase being greatest under wet weather conditions, permitting only poor aeration. On the day following cultivation after a period of wet weather, the amount of carbon dioxide in the soil air was reduced by over 90%. When the percentage of carbon dioxide in the soil air was at its highest, analyses of the air 12 in. above the surface showed only the normal content of carbon dioxide. C. T. Gimingham.

Chemical determinations to be made in the course of a soil survey. P. L. Gile (J. Amer. Soc. Agron., 1927, **19**, 285).—The composition of the colloidal and non-colloidal fractions is more significant than that of the whole soil; the mineralogical composition of the non-colloidal material is also important.
Chemical Abstracts.

Soil colloids. M. M. McCool (J. Amer. Soc. Agron., 1927, **19**, 289—297).—The soil colloids appear to control the chemical and physical reactions and properties of soils. During the weathering process the colloids, on account of their adsorptive capacity, act as protecting agents with respect to elements utilised for plant nutrition. If the results of the standard method of mechanical analysis are inaccurate, it is largely on account of incomplete dispersion of the samples when shaken.
Chemical Abstracts.

Determination of soil colloids. A. F. Joseph (Soil Sci., 1927, **24**, 271—274; cf. B., 1925, 730).—Comparison of the percentage of clay in a number of soils with the amount of colloids present, as determined by three different methods (heat of wetting, absorption methods, base-exchange), shows that, where there are important discrepancies between the two values, the content of colloid is larger than that of the clay. The properties on which the above methods for determining soil colloids are based are not peculiar to the colloid portion of the soil, but are possessed by such materials as silica, silt, and permutite. The most important properties of a soil which are due to inorganic colloids are known when the amount and nature of the clay fraction are known. C. T. Gimingham.

Composition of natural organic materials and their decomposition in the soil. I. Methods of determination of plant materials. S. A. Waksman and F. G. Tenney (Soil Sci., 1927, **24**, 275—283).—Analytical methods are given for the determination of those constituents of natural organic matter which are most important in influencing its rapidity of decomposition in the soil. The following determinations are made:—ether extract; organic matter soluble in cold water; lignin; cellulose; pentosans; protein insoluble in cold water; and ash. Analysis of plant materials by these methods accounted for 96—99·5% of the dry matter. Figures are also given for the more detailed analysis of the water-soluble and alkali-soluble fractions of the original material. C. T. Gimingham.

Chemical composition of soil organic matter as related to its effectiveness. F. J. Salter (J. Amer. Soc. Agron., 1927, **19**, 397—409).—Soil organic matter increases the solubility of some inorganic soil constituents, has buffer action, provides colloidal material, supplies nitrogen and mineral elements, and may have a carbon : nitrogen ratio which depresses the soil nitrates and favours or discourages the accumulation of humus.
Chemical Abstracts.

Effect of the method of sampling on the results of chemical analyses of horticultural plants. W. P. Tufts (Proc. Amer. Soc. Hort. Sci., 1925, **22**, 232—236).—In peach shoots taken at the same hour of the day the total nitrogen in the same class of tissue was practically constant over 12 days; the amino-nitrogen and carbohydrates fluctuated. The bark contained more nitrogen and carbohydrates than the wood. In apple and pear the composition, except as regards nitrogen, is uniform from apex to base; in the wood, the starch and reducing sugars decrease from apex to base.
Chemical Abstracts.

Determination of phosphoric acid in soils. A. Scheponovski (Engelhardt's Versuchs-stat., No. 2).—Sonnenschein's method is inaccurate. The soil is treated with 0·1*N*-potassium permanganate solution, acidified with sulphuric acid, and boiled; the residue is decomposed with oxalic acid, an aliquot part of the solution is precipitated with molybdate solution, the precipitate being dissolved in potassium hydroxide solution and titrated with sulphuric acid. Chemical Abstracts.

Total sulphur content of some cultivated soils. G. Bertrand and L. Silberstein (Bull. Soc. chim., 1927, [iv], **41**, 1380—1383).—The sulphur content, by the alkaline fusion method, of soils from seven localities in France varied from 0·202 to 5·175 g./kg., the soils richest in sulphur proving to be the most fertile. The importance to agriculture of a knowledge of the sulphur content of soils is thus established. A. R. Powell.

Field method for p_H determination [of soils]. M. F. Morgan (Ecology, 1927, **8**, 387—388).—An indicator solution, after percolation through the soil on a porcelain wedge, is compared with a colour chart.
Chemical Abstracts.

Lead arsenate and lime spray mixtures. P. A. van der Meulen and E. R. van Leeuwen (J. Agric. Res., 1927, **35**, 313—321).—Long-continued action of excess of calcium hydroxide on lead hydrogen arsenate results in the formation of basic calcium arsenate and hydrated lead oxide. The reaction occurs slowly and, under the conditions of normal spraying operations, probably less than 1% of the arsenate is decomposed. The action of atmospheric carbon dioxide on a mixture of calcium hydroxide and lead hydrogen arsenate does not produce an appreciable increase of "soluble arsenic" until after all the hydroxide is carbonated. The addition of slaked lime to lead arsenate spray fluids prevents injury to tender foliage by "soluble arsenic."
C. T. Gimingham.

Bog hay. R. E. Evans (Welsh J. Agric., 1927, **3**, 119—147).—Hays chiefly composed of *Molinia*, sedges, sheep's fescue, or bent, respectively contain more protein than those composed of *Nardus*, tufted scirpus, or rushes. Bog hay contains 2·06—4·47% of silica-free ash; meadow hay contains 6·76%. Phosphate is high in hay

composed chiefly of rushes or bent, and low in hays composed of cotton sedges or tufted scirpus. Typical bog herbage is poor in iron and chlorine in comparison with meadow hay; early-cut hay is richer in iron and chlorine and has a higher nutritive value. Ammonium sulphate and kainite had less effect than basic slag on the yield and chemical composition of the natural herbage of peaty soils. CHEMICAL ABSTRACTS.

Electrodialysis of soils and the Mattson cell. N. A. CLARK, H. HUMFELD, and A. O. ALBEN (Soil Sci., 1927, **24**, 291—295; cf. Mattson, B., 1927, 22).—The technique employed in working with the cell devised by Mattson for the electrodialysis of soils is discussed and some experiments on the rate of extraction under various conditions are recorded. Variations in the electrodes in order to use less platinum and the introduction of a cooling system into the cell are suggested.

C. T. GIMINGHAM.

Determination of nitrogen. SHEDD.—See XIX.

XVII.—SUGARS; STARCHES; GUMS.

Simultaneous adsorption of sucrose and colouring matters by carbons. J. VAŠATKO (Z. Zuckerind. Czechoslov., 1927, **52**, 45—52).—Experiments are described showing that an increase of the sucrose adsorption in the presence of colouring matters is accompanied by an increase in the capacity of the carbon to take up water from the solution. In practice, it follows that the conditions for the best colour adsorption are a dilute solution and a high temperature.

J. P. OGILVIE.

Determination of the amount of sulphur dioxide in Mauritius direct-consumption sugars. L. BAISSAC (Int. Sugar J., 1927, **29**, 538—539).—Direct titration by iodine of a solution of the white sugar (100 g.) in 300 c.c. of water with 10 c.c. of 30% sulphuric acid gave results agreeing very closely with those found by the distillation method. By applying this direct procedure to fifty-five samples of Mauritius white sugar, fifty were found to have a sulphur dioxide content of 10—70 p.p.m., and the remaining samples (grade B), which are not direct-consumption sugars, contained 72—122 p.p.m. J. P. OGILVIE.

Measurement of colour [of sugar liquors] in Stammer units on a Kober-Klett colorimeter. K. S. RITCHIE (Ind. Eng. Chem., 1927, **19**, 1289—1290). —Any Duboscq-type colorimeter may be used to determine the colour of sugar and molasses liquors in Stammer units. The standard colour solution chosen was ferric chloride containing 62 g. of pure iron in 300 c.c. of solution, and reproducible results were obtainable over a period of two years. It was found, however, that, unlike caramel solutions, the ferric chloride dilution curve does not follow Beer's law, *i.e.*, the colour intensities of two solutions are not exactly the same when the products of the depth and concentration of each are equal. With a 3 mm. column in the particular colorimeter used, the Stammer " depth constant " was found to be 0·369 mm. Illustrations of the method of using this figure are given. E. HOLMES.

Improvement of the Stammer colorimeter [for sugars]. K. ŽERT (Z. Zuckerind. Czechoslov., 1927, **52**, 57—63).—Normal clarified juices required a colour-glass composed of yellow, red, and green in the proportions of 80 : 27 : 8, whilst for molasses and after-products the proportions are 80 : 42 : 16. Examination of colour-glasses of the Stammer instrument and those of similar type shows their colour components to be often very variable. It is preferable to match each of the colour components separately, using a bluish-violet filter for the yellow, a green for the red, and a red for the green. To obtain compensation in the two halves of the field with cloudy solutions, the opening admitting light to the glasses is slightly screened, while the height of the juice column is adjusted at the same time.

J. P. OGILVIE.

Determination of carbonate in syrups etc. I. B. MINTZ and Y. I. ZILBERMAN (Nauch. Zapiski [Russia], 1927, **5**, 45—46).—The sample (2—3 g.) is slowly heated with 1 c.c. of 14% oxalic acid solution, and ashed over a small flame; the carbon may be removed in a muffle.

CHEMICAL ABSTRACTS.

Sucrose crystallisation. I. A. KUKHARENKO and A. K. KARTASHEV (Nauch. Zapiski [Russia], 1927, **4**, 369—371).—Calcium chloride is a strong positive paralyser of crystallisation. CHEMICAL ABSTRACTS.

Storing sugar beet by drying. G. S. BENIN (Nauch. Zapiski [Russia], 1927, **4**, 253—266).—Drying for 1 hr. causes a loss of 0·1% and for 12 hrs. a loss of 1·82% of sugar; apparently some of the invert sugar is decomposed. Drying may be effected above 100°.

CHEMICAL ABSTRACTS.

PATENTS.

Treatment of liquids [coagulation of the colloids of sugar juices]. GILCHRIST & Co., Assees. of W. C. GRAHAM (E.P. 247,542, 25.1.26. U.S., 13.2.25).—A rapidly circulating supply of the juice is subjected to the action of an alkaline reagent distributed, in finely-dispersed form, continuously and at a substantially uniform rate over the surface of the liquid in rapid circulation within itself and within a single chamber, to avoid local over-neutralisation at any point and to effect progressive change in its p_H value. The temperature of the juice is suitably regulated during the operations involved, and is preferably maintained approximately at that at which the juice under treatment enters the apparatus. J. P. OGILVIE.

Treatment of bagasse. H. ISTELLI (U.S.P. 1,645,242, 11.10.27. Appl., 24.12.25).—Bagasse is agitated with an extracting medium and is simultaneously sprayed therewith. The resulting solution of sugar is then removed. D. J. NORMAN.

Evaporation of sugar solutions (E.P. 265,127).—See I.

Saccharification of wood (E.P. 278,450).—See V.

XVIII.—FERMENTATION INDUSTRIES.

Lambic fermentation. M. H. VAN LAER (Bull. Féd. Ind. Chim. Belg., 1927, **6**, 357—365, 413—425).—The brewing of lambic beer by the artificial seeding of the wort is described. The following four groups of organisms are concerned in the lambic fermentation:

a *Saccharomyces* species, *S. Bruxellensis*, *Brettanomyces Bruxellensis*, *Brettanomyces lambicus*, and *B. viscosus Bruxellensis*. The flavour produced by *Brettanomyces Bruxellensis* is sharp and pleasant, and is preferred to the mouldy flavour due to *Brettanomyces lambicus*. The *Brettanomyces* give a slow fermentation with a high acidity. In the presence of *S. Bruxellensis* this acidity is decreased, but the percentage attenuation is increased. In anaerobic life the production of acidity almost vanishes, and advantage is taken of this fact to fix the amount of acidity by choosing the moment for bottling. The presence of *B. viscosus Bruxellensis* allows the formation of the acidity to proceed slightly further, even in the absence of air, and thus facilitates the maturation in bottle. The best results are obtained by seeding chilled wort with a mixture of *S. Bruxellensis*, *Brettanomyces*, and the *B. viscosus Bruxellensis*. Compared with the lambic beer allowed to seed naturally, the artificially seeded worts ferment more quickly and eventually give a greater percentage of attenuation and higher final acidities. They clarify quickly, and can be bottled after a fermentation lasting four or five months. They have the typical lambic quality, drink older and fuller, and do not have the mouldy flavour which is an objection to the natural lambic. C. Ranken.

Absorption of odours by beer. A. J. Murphy and F. A. Mason (Bull. Bur. Bio-Tech., 1927, **2**, 165—171).—Beers can acquire a flavour by the mere absorption of odour without actual contact with the odoriferous material and without biological infection. When a small bulk of wort is fermented and racked in an atmosphere permeated by each odour under consideration, no odour or flavour is communicated to the beer by phosphorus, carbon disulphide, hydrogen sulphide, mineral naphtha, and sulphur dioxide. On the contrary, free chlorine, gas tar, coal-tar disinfectant, and mould communicate their characteristic odours and flavours to the beer, whilst the flavour derived from exposure to turpentine is of a decidedly fruity nature. A metallic-looking film appears upon the surface of the beers in contact with chlorine, whilst those fermented in the presence of hydrogen sulphide, mineral naphtha, and gas tar are inclined to be hazy. C. Ranken.

Vanillin in wine distillates, brandies, and adulterated brandies. G. Reif (Z. Unters. Lebensm., 1927, **54**, 90—101).—Wine distillates contain substances other than vanillin which give similar colour reactions, and satisfactory analytical results are obtained only when the vanillin is isolated by sublimation. The bromine–ferric sulphate method of Mork and the phosphotungstic–phosphomolybdic acid test of Folin and Denis are recommended for this purpose. The brown colour of wine distillates depends on the presence of phenolic compounds therein. Vanillin in wine distillates is extracted from the wooden casks used for storage and its quantity depends on the wood surface exposed, the time of storage, and to a certain extent on the amount of alcohol in the distillate. The amounts of vanillin extracted from the wood per litre of liquor averaged 0·2 mg. for distillate and 0·1 mg. for brandy. Adulterated brandies contained no vanillin.
A. G. Pollard.

Adulteration of brandy and its detection. G. Graff (Z. Unters. Lebensm., 1927, **54**, 102—136).—Extensive and detailed analyses of wines and spirits are recorded. The proportions in which wines and distillates are mixed in the manufacture of brandy vary so widely that detection of adulteration is exceedingly difficult. Various methods for this purpose are examined and discussed, and suggestions for possible new methods are recorded. An examination of the higher alcohols in distillates and brandies appears to be of value in this connexion. A. G. Pollard.

Determination of total solids in malt vinegar. J. F. Laudig (J. Assoc. Off. Agric. Chem., 1927, **10**, 520—521).—On account of the retention of acetic acid by the malt vinegar solids in amounts varying according to the daily fluctuations in relative humidity and atmospheric pressure, differences as high as 0·8% were found when determinations of total solids were made on the same sample upon different days by the A.O.A.C. method. The differences with cider vinegar were negligible. Lower but consistent results were obtained by making three evaporations to dryness instead of two, the solids being afterwards dried in a steam oven for 2½ hrs.
F. R. Ennos.

Regeneration of fats from the wastes in yeast manufacture. M. A. Bendecky (Ukraine Chem. J., 1926, **2**, 136—138).—Fats (*e.g.*, a mixture of lard and beef fat) which are used in the manufacture of yeast for the destruction of the foam formed during fermentation and are not absorbed by the yeast, but are left on the walls of the vessels at the end of the process, showed on analysis of the waste products the presence of 68·3—75·2% of fats. These fats are regenerated by boiling for 10—15 min. with twice the quantity of finely-powdered salt necessary for saturation of the water present in the waste products. The excess of salt forms with water and organic substances present a thick mass, from which the fat can be poured off and used again. The fats so regenerated have a higher m.p., a lower iodine value, and a higher acid value than those used as starting materials. A. Ratcliffe.

Patents.

Manufacture of yeast. Vereinigte Mautner'sche Presshefe-Fabr. G.m.b.H., and E. Fould-Springer (Austr. P. 105,784—5, [a] 20.9.19 and [b] 29.11.20).—(a) Nutrient solutions of higher, lower, or equal concentration, according to the calculated concentration of the fermenting liquid, are led continuously or at suitable intervals into a fermenting liquid containing sugar or other suitable raw material for the propagation of yeast with or without the formation of alcohol. The concentrations are so regulated that the most favourable life conditions for the yeast are present continuously. (b) A more concentrated wort is added to a dilute wort or mash in order to replace losses due to yeast reproduction. The concentrations are so regulated that considerable amounts of alcohol are formed, which may or may not be separated later during the process. C. Ranken.

Clarification of white wine. Anc. Maison Gerbaud Soc. Anon. (F.P. 619,858, 14.12.25).—The wine is heated with washed powdered glass in the absence of air. C. Ranken.

XIX.—FOODS.

Milk supply of towns. F. E. Nottbohm (Z. Unters. Lebensm., 1927, **54**, 201—212).—The effect of many factors—physiological, pathological, and mechanical—on the chemical composition of milk is discussed in the light of the observation of food regulations. The period which should elapse to allow of the elimination of the colostrum before utilising the milk for human consumption is examined from the point of view of chemical analyses. Higher proportions of fat and protein and inferior palatability are characteristic of colostral milk. During periods of " heat " the fat content of the milk of some cows increases. The milk of the majority of animals, however, is unaffected. Variations in the solids-not-fat of milks show a close periodicity year by year with well-defined minima in April and July—August. General but slight differences in the composition of milk from different quarters of the udder were observed—that of corresponding pairs being similar. Milk from a diseased quarter differs from that of the three others in being usually of lower density and having decreased fat, dry matter, and sugar, but increased chlorine content. The later portions of milk from any one milking have slightly higher fat content. Evening milk has more fat than that of the morning or mid-day. Shortening of the period between milking tends to increase the fat content.

A. G. Pollard.

Vitamins. XVI. Vitamin-*A* in dried milks made by vacuum and aeration methods. R. A. Dutcher, H. E. Honeywell, and C. E. Dahle (J. Biol. Chem., 1927, **75**, 85—94).—Dried milk, whether prepared by the vacuum method or by aeration, is deficient in vitamin-*A* as compared with raw milk, the loss being greater in the aeration process, and still greater after sterilisation. C. R. Harington.

Adulteration of sulphuric acid to increase Babcock test reading [of milk]. W. E. Petersen (J. Dairy Sci., 1927, **10**, 261—262).—Aqueous soap solution (Palmolive china soap 1 pt., water 2 pts.) (6 c.c.), followed by 16 c.c. of fat-saturated benzine, are added to 400 c.c. of sulphuric acid (*d* 1·83). The mixture is stable, and gives an increased reading of 0·8%.

Chemical Abstracts.

Rate of acid production in heated milk. E. O. Whittier and A. G. Benton (J. Dairy Sci., 1927, **10**, 343).—A curve for practical use is based on data previously published. Chemical Abstracts.

Annatto extract for colouring butter. T. L. Rao (Madras Agric. Dep. Yearbook [1925], 1926, 1—6).—The crude dye (10·25%), containing much mucilaginous matter, is obtained by cold-water fermentation followed by maceration of the seed, decantation, and washing. Pure dye (7%) is obtained by extraction with 1% sodium carbonate solution, followed by acidification. Aqueous ammonia is a good solvent.

Chemical Abstracts.

F.p. of cream, and detection of added water. F. J. Doan (J. Dairy Sci., 1927, **10**, 353—369).—Cream and skim-milk have the same f.p. (average —0·55°) as the original milk. A modified formula is given whereby the amount of water added to the original whole milk can be calculated if the weight or volume of both cream and skim-milk portions are known.

Chemical Abstracts.

Presence of arsenic, lead, and copper in fruit and fruit products as a result of spraying. K. Lendrich and F. Mayer (Z. Unters. Lebensm., 1927, **54**, 137—159).—Modifications in the analytical technique for the determination of arsenic are described. Australian apples and pears examined were free from lead and arsenic, but occasional traces of copper were observed. American apples commonly contained all three poisons. On apples from the Western States lead and arsenic were more common than copper, but on those from the Eastern States copper was more general. The proportions in which lead and arsenic were found together varied widely. On grapes grown in Germany all three poisons were common. Arsenic and copper but no lead could be found in wines produced from sprayed grapes.

A. G. Pollard.

Food value of the nut of *Trapa bispinosa*. B. B. Brahmachari and N. K. Chatterjee (Indian Med. Gaz., 1927, **62**, 365—370).—The nut contains: water 84·58, proteins 2·37, fats 0·18, starch 9·14, soluble carbohydrates 2·25, fibre 0·56, ash 0·92%. The ash contains K_2O 9·7, Na_2O 19·9, CaO 2·5, MgO 1·7, Fe_2O_3 0·9, MnO 0·5, P_2O_5 25·1, SO_3 13·1, $CaCl_2$ 3·6, SiO_2 0·8%. Chemical Abstracts.

Rapid boiling as an aid to a shortened period of digestion in the determination of nitrogen. O. M. Shedd (J. Assoc. Off. Agric. Chem., 1927, **10**, 507—520).—By the use of a grid burner of the Fisher or Meker type it was found possible to shorten the period of heating with sulphuric acid etc. in the Kjeldahl–Gunning–Arnold method to 20 min., the whole process being completed in 1—1¼ hrs. From a comparison of the short and the regular 5 hr.-digestions made on a variety of feedstuffs, fertilisers, soils, etc., it appears that, except in the case of pyridine, the short-digestion process gives results in agreement with the longer official method provided that mercury is used as the catalyst. Although copper is as effective a catalyst as mercury in the long process it does not give reliable results in the short process. The best conditions advised for the distillation are the use of the minimum quantity of sodium hydroxide required to liberate the ammonia, the minimum quantity of zinc (not more than 100 mg.) to prevent bumping, and the precipitation of copper or mercury by addition of potassium polysulphide, which gives an even-boiling solution and also retards the carrying over of sodium hydroxide as a spray.

F. R. Ennos.

Freezing points of cheeses. P. D. Watson and A. Leighton (J. Dairy Sci., 1927, **10**, 331—334).

Detection of boron. Holmes.—See VII.

Iodine in beverages. Settimj.—See XXIII.

Patents.

Preparation of live-stock feeds. H. A. Gill. From U.S. Farm Feed Corp. (E.P. 278,818, 20.7.26).—Farm roughage is converted into a relatively highly nutritive and digestive feeding stuff for live-stock by mixing the moist, partly compressed material with

magnesium or calcium hydroxides, sodium chloride, or sulphur, and subjecting it to the action of ferment-containing agents inherent therein and/or of added ferment-containing agents, *e.g.*, malt or yeast, with ready access of air. The product may be given a higher feeding value by means of calcium phosphate, and its palatability increased by a small proportion of benzaldehyde, both being added before the fermentation process.

F. R. Ennos.

Preparation of egg products [for use as emulsifiers]. A. K. Epstein (E.P. 279,159, 19.7.26).—See U.S.P. 1,595,765—6; B., 1926, 896.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Production of formaldehyde by oxidation of hydrocarbons. W. Ledbury and E. W. Blair (Chem. Res., Spec. Rep. No. 1, Dept. Sci. Ind. Res., 1927, 54 pp.).—The report contains the results of typical experiments and a summary of work previously published on the production of formaldehyde by the controlled oxidation, under varying conditions, of ethylene, methane, coal and coke-oven gas, and hexane, also on the partial formaldehyde vapour pressures of aqueous formaldehyde solutions, the bisulphite and ammonia compounds of formaldehyde, and investigations of the distillation of formaldehyde solutions at atmospheric pressures. Distillation of formaldehyde solutions under pressure gives a distillate having a higher concentration of formaldehyde than that obtained by distillation under atmospheric pressure and, at the optimum pressure of 60 lb./sq. in., dilute formaldehyde solutions give distillates having approximately three times the strength of the initial solutions. The results obtained with small and semi-technical-scale continuous processes are described, and the costs of the preparation of 40% formaldehyde solutions on a commercial scale are outlined. Examination of the action of formaldehyde on metals at high temperatures and pressures shows that whilst nickel, aluminium, and copper had only a slightly deleterious effect, iron and Monel metal caused considerable decomposition of the formaldehyde. The economic application of the above methods for the large-scale production of formaldehyde is fully reviewed, and proposed plants are described.

E. H. Sharples.

Analyses of mixtures of water, methyl alcohol, and ethyl alcohol. E. Berl and L. Ranis (Ber. 1927, **60**, 2225—2229).—The method depends on the determination of the density and refractive index at 17·5° of mixtures of water, methyl alcohol, and ethyl alcohol obtained by distillation of unknown mixtures. The third factor necessary for the determination of the three unknowns is given, since the weight of the solution is equal to the sum of the weights of the three components.

H. Wren.

Determination of nicotine in tobacco and tobacco smoke. B. Pfyl and O. Schmitt (Z. Unters. Lebensm., 1927, **54**, 60—77).—The determination of nicotine by the usual methods is complicated in the case of denicotinised and treated tobaccos by the presence of other bases produced by fermentation, oxidation, etc. during the denicotinising process. To avoid the resulting inaccuracies nicotine is separated by steam distillation and precipitated in neutral solution as dipicrate. The precipitate after filtration and washing is titrated with sodium hydroxide solution against phenolphthalein in the presence of toluol. As a check on the amount of free base present a portion of the toluol solution of nicotine is titrated with iodoeosin. Apparatus is described for trapping the nicotine and other bases in tobacco smoke. In the subsequent distillation of nicotine from the liquor excess of soda is avoided and the determination carried out as above. The smoke of "harmless" and "nicotine free" tobaccos contained as much nicotine as the ordinary qualities. The rate of burning has considerable influence on the quantity of nicotine in tobacco smoke.

A. G. Pollard.

Iodometric evaluation of Methylene Blue. W. C. Holmes (J. Assoc. Off. Agric. Chem., 1927, **10**, 505—507).—In the A.O.A.C. method for the determination of Methylene Blue in medicinal samples based on the iodine absorption of the dye when precipitated in the presence of acetic acid by a large excess of iodine, the amount of iodine absorbed by the dye diminishes with the concentration of acid present and increases with that of the residual iodine in the solution. For accurate work the official method should be calibrated for variations in dye concentration.

F. R. Ennos.

Assay of sulphonal tablets. L. E. Warren (J. Assoc. Off. Agric. Chem., 1927, **10**, 523—526).—In the absence of other chloroform-soluble substances, the sulphonal content may be determined by extracting the powdered tablets with chloroform either by repeated maceration and filtration or by means of a Bailey or a Soxhlet extractor. The extract is evaporated at ordinary temperature by the aid of a gentle current of air, and the residue dried to constant weight over sulphuric acid.

F. R. Ennos.

Activity of digitalis preparations. VI. Chemical methods of assay of digitalis preparations. C. de Lind van Wyngaarden (Arch. exp. Path. Pharm., 1927, **126**, 135—142. Cf. Knudson and Dresbach, A., 1922, ii., 882).—The colorimetric method described by Knudson and Dresbach (*loc. cit.*) for the assay of digitalis and strophanthin preparations does not give accurate results. The method of Fromme and Menlenhoff for the assay of strophanthin preparations yields systematically high values.

W. O. Kermack.

Analysis of jalap. H. E. Dale (Pharm. J., 1927, **119**, 516—517).—The resin content is determined by extracting the powdered material with hot alcohol, filtering, evaporating an aliquot portion of the filtrate to dryness, and washing with successive specified quantities of water at 65°. The washed resin is taken up again in alcohol, transferred through a filter to a tared vessel, the solution evaporated, and the residue weighed.

S. I. Levy.

Physical chemistry of dental cements. W. S. Crowell (J. Amer. Dental Assoc. 1927, **14**, 1030—1048).—A description of the composition of dental cements and of the reactions which occur on spatulation. The fluorine in silicate cements is insoluble and therefore non-toxic. In 15 min. after mixing, the acidity is 1 in

3×10^4 or less; after 7 days it reaches p_H 6·5 in zinc phosphate cements or p_H 5·5—5·8 in silicate cements.
Chemical Abstracts.

Oxidising and auto-oxidising power of turpentine oil. G. Testoni (Ind. saponiera, 1927, 27, 111—113).—A number of colour reactions are described which show that the oxidising power of turpentine oil is similar to that of many oxidising agents. The colour developed by benzidine in the presence of a trace of acetic acid should be applicable to the determination of traces of oxidising agents. Unlike Greek oil, American, French, Austrian, and Russian oils, pine oil, regenerated turpentine, camphor oil, hexalin, and heptalin do not react with quinol, pyrogallol, or benzidine, but only with *p*-phenylenediamine. The auto-oxidation of turpentine furnishes a solid, monobasic, saturated acid, probably a polymeride.
Chemical Abstracts.

Composition of the essential oil of *Pistacia mutica*, Fisch. et Mey. N. Y. Demyanov and V. I. Nilov (Zapiski Gosudar. Nikit. Opit. Bot. Sada [Crimea], 1926—7, 9, No. 1, 49—62).—The oil contains *d*-α-pinene, *i*-β-pinene, *l*-limonene, and a substance, $C_{10}H_{16}O$, reminiscent of myrtenol or pinocarveol.
Chemical Abstracts.

Composition of the essential oil of *Siler trilobum*, Scop. N. Y. Demyanov and V. V. Williams (Zapiski Gosudar. Nikit. Opit. Bot. Sada [Crimea], 1926—7, 9, No. 1, 62—70).—The oil contained perillaldehyde, *d*-limonene, and a hydrocarbon, *d* 0·8501, [α] —87·87°, *n* 1·4722.
Chemical Abstracts.

Ethereal oils of Crimean plants. V. I. Nilov and V. V. Williams (Zapiski Gosudar. Nikit. Opit. Bot. Sada [Crimea], 1926—7, 9, No. 1, 3—49).—Data are recorded for the oils of 60 Crimean plants.
Chemical Abstracts.

Ethyl alcohol from vegetable matter. Carrière.—See V.

Analysis of mixtures of aliphatic nitrates. Rinkenbach.—See XXII.

Patents.

Recovery of concentrated acetic acid and other products originating in the carbonisation of wood. H. Suida (E.P. 275,158, 21.2.27. Austr., 31.7.26).—Volatile products from a wood-carbonising retort are freed from tar by washing with tar oils, and the acetic acid is extracted as vapour with a substance insoluble in water and having a higher b.p. than acetic acid, *e.g.*, cresol, and is separated into acetic acid and extraction agent in a vacuum apparatus. The volatile products leaving the extraction column are subjected to partial condensation to separate water and any entrained extraction agent, and then to further condensation to separate wood spirit.
W. G. Carey.

Manufacture of valuable organic compounds. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 278,777, 10.6.26).—The oily mixtures of oxygen-containing organic compounds of a higher order than methyl alcohol obtained in the catalytic hydrogenation of carbon oxides (cf. E.P. 227,147, 229,714, 237,030, and 238,319; B., 1925, 189, 338, 784, and 900), which are often coloured and unpleasant smelling, are converted by catalytic hydrogenation in the vapour state into colourless oils of pleasant or slight odour, and consisting substantially of alcohols separable by fractionation. Hydrogenation is carried out at 100—180°, below the b.p. of the oil, with hydrogen free from carbon oxides, using as catalysts nickel, cobalt, copper, platinum, etc., alone or mixed, with or without a support and/or activating mixture.
B. Fullman.

Preparation of gall acids. Chem. Fabr. vorm. Sandoz (E.P. 269,925, 22.4.27. Ger., 24.4.26).—Gall acids free from fatty acids and colouring matter are obtained by adding the crude acids, yielded by acidification of the hydrolysed gall as usual, to a well-stirred mixture of a fat solvent (toluene) and a solution of a salt of a weak acid (sodium acetate, sulphite, or cholate, calcium carbonate) and warming. The colouring matter and fatty acids are removed by the fat solvent, leaving the gall acids as a crystalline powder, yielding on treatment with alcohol 75% of nearly pure cholic acid, from which is obtained on crystallisation etc. a product having m.p. 196—197°. The use of the salt of a weak acid as above described is obviated by precipitating the crude gall acids from the hydrolysed gall in the presence of such a salt, either added, or formed by neutralising part of the alkali used in the hydrolysis by a weak acid; or by precipitating the acids direct while avoiding any excess of mineral acid, even locally. In either case, the aqueous suspension of the crude product is then extracted with a fat solvent.
B. Fullman.

Extraction of marine algæ. P. Gloess ([a] F.P. 578,564, 30.3.23, and Addn. Nos. [b] 28,596, 12.9.23, [c] 28,717, 16.10.23, [d] 31,475, 24.8.25).—(a) Highly viscous alginate solutions are obtained by extracting algæ with a counter-current of water, and periodically expressing the water from the material. The aqueous extract contains alkali or magnesium chlorides and sulphates and an organic iodine compound which can be separated as its lead or copper compound. The alginate solutions can be used (b) for binding sand, sawdust, etc., or mixtures of these with lime or magnesia, or (c) for the preservation of perishable foodstuffs, such as fish, meat, or fruit, or (d) as flocculating agent in the purification of water, for purifying soaps, or for the manufacture of plastic material, photographic films, etc.
L. A. Coles.

[Solvent for use in] varnishes, shoe-creams, etc. I. G. Farbenind. A.-G. (E.P. 275,652, 15.12.25. Ger., 23.12.24).—Fats, waxes, solid hydrocarbons, natural or artificial resins, etc. are dissolved in 1 : 4-dioxan and, if desired, other solvents. Dyestuffs may be introduced.
S. S. Woolf.

Production of stable, sterilisable, complex aurosodium thiosulphate solutions. L. Benda, Assr. to I. G. Farbenind. A.-G. (U.S.P. 1,648,213, 8.11.27. Appl., 30.11.25. Ger., 27.1.25).—See E.P. 246,809; B., 1927, 252.

Manufacture of quinine solutions suitable for subcutaneous injections. A. Liebrecht, Assr. to Chem.-Pharm. Corp., Bad Homburg (U.S.P. 1,648,487, 8.11.27. Appl., 13.7.25. Ger., 14.7.24).—See G.P. 427,668; B., 1926, 853.

Methane (F.P. 619,621).—See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Modified sulphide-nuclei theory of [photographic] sensitivity. J. SOUTHWORTH (Brit. J. Phot., 1927, **74**, 641—642).—The suggestion that there is a certain minimum size of development nucleus necessary to be effective in development is considered to be unwarranted and unnecessary. Whether or not a nucleus is effective in development is considered to be determined by the degree of its contact with a grain. The fact that the sensitising nuclei of silver sulphide in an unexposed emulsion do not give rise to fog is ascribed to their being not sufficiently closely related to the silver halide. The effect of light is possibly to bring the nuclei into effective contact with the grains, although the explanation is more likely to be found on the lines of the halogen-absorption theory of sensitivity. The maximum of sensitivity is assumed to occur when the silver sulphide nuclei are related to the silver halide as closely as possible, short of being so close as to cause fog. When a fully ripened emulsion is made acid, speed is reduced because the nuclei are then not so closely related to the silver halide. If the emulsion is then made alkaline again, the speed is restored owing to the closer approach of the nuclei. An alkaline emulsion heavily fogged with excess of thiocarbamide yielded less fog on being made acid.

W. CLARK.

Fog correction of photographic densities: a sensitometric study. H. A. PRITCHARD (Phot. J., 1927, **67**, 447—459).—By studying the growth of fog for long development times after development of the image density was practically complete, more consistent sets of corrected sensitometric data were obtained than by previous methods of fog correction. The Wilsey fog correction, based on the assumption that the mass of silver developing as fog is proportional to the mass of silver unaffected by exposure, neglects several factors which have an appreciable effect unless the fog is small. The Wilsey method appears to give satisfactory results provided the image density can be practically completely developed before the fog density reaches a value of 0·5. Study of Bloch's method of measuring fog in a small protected area in the centre of each density showed that the influence of reaction products on development is practically negligible with the metol–quinol *X*-ray developer used. By prolonged development it is possible to develop all the silver in the emulsion as fog, but the density of maximum fog is considerably less than the maximum image density. Reversal in the lower exposures observed on prolonged development of positive film persists at complete development of all the silver in the film. The large variations in photometric equivalent are thought to contribute considerably to the failure of the Wilsey fog correction under extreme conditions.

W. CLARK.

PATENTS.

Preparation of [photographic] printing paper. E. GAY (F.P. 616,562, 20.10.25).—Paper or other base is treated with a solution of a diazo compound of anisidine or naphthanisidine, or their halogen, nitro-, sulpho-, carboxy-, or other derivatives, alkylated or arylated in the hydroxyl group. On exposure the unprotected parts are bleached by decomposition of the diazo compound, and on development with an alkaline solution of H-acid a violet positive image is formed.

W. CLARK.

Highly sensitive photographic plates and films. P. SPECKLIN (F.P. 615,932, 27.9.25).—Substances, such as small amounts of barium or calcium tungstate, which fluoresce under the influence of *X*-rays, are incorporated in the sensitive coating.

W. CLARK.

Preparation of coloured images on paper. V. WEIL (G.P. 442,535—6, [A] 28.3.26, [B] 12.10.24).—(A) The emulsion on a sensitive paper is coated with a non-uniform colour layer of high transparency which is not attacked by the developer or fixing bath. The colour film is prepared by any convenient printing process or by spraying with coloured collodion. (B) A coloured mosaic screen is used over the photographic layer on a white base in such a way that very fine capillary spaces are left between the points on the screen. The developer and other solutions can penetrate to the emulsion through the fine capillaries and spread out under the dots of the screen. The colour above the developed silver practically disappears, whilst that over the undeveloped silver remains visible owing to the white base.

W. CLARK.

Manufacture of photographic copies by reversal. I. G. FARBENIND. A.-G., Assees. of A. MILLER (U.S.P. 1,632,740, 14.6.27. Appl., 23.9.25. Conv., 21.3.25).—A freshly developed silver negative is treated with a solution of ferric chloride and potassium ferricyanide, the deposited silver being rapidly coated with Prussian Blue. The remainder of the negative is mordanted by the treatment, so that, on washing, the deposit is removed to the mordanted portion and a reversed positive is obtained.

T. S. WHEELER.

Toning and fixing of photographic prints. E. DEVIENNE (E.P. 272,925, 15.6.27. Fr., 21.6.26).—A single, stable toning bath for print-out images consists of a solution of thiosulphate to which are added salts of iron, copper, or chromium. Suitable salts are: ferric or chromic salts, in which case a convenient formula is sodium thiosulphate, 20 g., ferric sulphate 2 g., water 100 c.c.; or ferrous salts, iron compounds containing the iron in the acid radical (*e.g.*, ferrocyanide or ferricyanide), or copper salts. In the second group, acid is also added to the bath. Alkali thiocyanate may be added, especially in the case of ferrous or copper salts, to stabilise the solution. A suitable bath is sodium thiosulphate 20 g., copper sulphate 2 g., ammonium thiocyanate 1·5 g., water 100 c.c., and dilute sulphuric acid (10% by vol.) 3 c.c.

W. CLARK.

Ammonia development of [photographic] prints. H. WINKLER and H. VOLKMANN (G.P. 441,888, 13.11.25).—In order to accelerate the time of development, the prints are carried between endless bands, and treated with artificially vaporised or dispersed ammonia solution.

W. CLARK.

[Two-part rotatable screen for use in] natural colour cinematography. "CHROMO" FILMGES.M.B.H. (E.P. 274,804, 27.11.26. Austr., 22.7.26).

XXII.—EXPLOSIVES; MATCHES.

Analysis of mixtures of aliphatic nitrates by means of the refractometer. W. H. Rinkenbach (Ind. Eng. Chem., 1927, **19**, 1291—1292).—Since no chemical test is available for detecting nitroglycerin in the presence of nitrated glycols, an examination of their refractive indices has been made. Tables are given for the refractive indices of nitroglycerin, ethylene glycol dinitrate, diethylene glycol dinitrate, 70% nitroglycerin-30% nitropolyglycerin, and varying mixtures of ethylene glycol dinitrate and nitroglycerin, at temperatures of 15°, 20°, 25°, and 30°. The values at 15° give the smoothest curves, so that this appears to be the optimum temperature at which to work. This method of analysis offers a fair degree of accuracy when examining the pure substances. The effect of small quantities of oils, fats, and resins is discussed. E. Holmes.

Apparatus for studying the ignition process of inflammable gas-air mixtures by explosives. G. St. J. Perrott and D. B. Gawthrop (Ind. Eng. Chem., 1927, **19**, 1293—1295).—An explosion gallery (having a series of plate-glass windows) and a moving film camera are described, with which it has been found possible to record the phenomena occurring when varying amounts of explosives are fired into an atmosphere containing 8% of natural gas. Examples are given of the actual flame propagation photographs, together with detailed analyses of the various features, as flame length and duration. The method is expected to lead to correlation between the empirical "gallery test" of "permissible" coal-mine explosives and the fundamental factors—flame temperature, rate of detonation, and pressure. E. Holmes.

XXIII.—SANITATION; WATER PURIFICATION.

Microchemical investigations of the iodine in beverages and foods. M. Settimj (Annali Chim. Appl., 1927, **17**, 426—432).—The proportions of iodine, determined by Fellenberg's method (A., 1925, i, 329), in the various potable water supplies of Rome are given. No inverse proportionality between hardness and iodine content is observed, and Fellenberg's statement that hard waters containing much lime and magnesia lose their iodine rapidly is not confirmed. In some instances the method requires modification; with the Trevi water, addition of potassium carbonate is superfluous, and with the Lancisiana water, which contains considerable proportions of nitrates, reduction of these by means of zinc dust is found necessary. T. H. Pope.

Micro-determination of iodine in potable waters. M. Settimj (Annali Chim. Appl., 1927, **17**, 432—445).—With certain modifications, Fellenberg's method (A., 1925, i, 329) gives satisfactory results when applied to the determination of iodine in water, but when the iodine is present in very small proportion, further modifications are necessary to render the method sufficiently sensitive. Repeated heating of the organic matter is essential and, especially when small amounts of iodine are concerned, Steffen's method (B., 1926, 902) is not recommended. T. H. Pope.

Comparison of the results of Clemesha's method and the test of citrate utilisation as applied to water supplies in Burma. J. Taylor [with C. de C. Martin, J. V. R. Naidu, and P. N. R. Naidu] (Indian J. Med. Res., 1927, **14**, 801—836).—Clemesha's method gave results which most nearly approximated to the sanitary circumstances of the sources of the water samples tested. The combined use of the citrate, methyl-red, and Voges–Proskauer tests gave valuable information supplementary to the Clemesha classification. Chemical Abstracts.

Colorimetric method for the field determination of the carbon dioxide tension and free carbon dioxide, hydrogen carbonates, and carbonates in natural waters. I. Theoretical. E. B. Powers (Ecology, 1927, **8**, 333—338).—The hydrogen-ion concentration is determined before and after aeration with air of known carbon dioxide tension, whence n in the equation $C_H = (KkP)^n$, where K (a constant) is $3 \cdot 04 \times 10^{-7}$, and k, the partial pressure of the carbon dioxide under standard conditions (P), is 0·04415, may be determined. Thus the original value of P is computed. Chemical Abstracts.

Improved micro-Kjeldahl ammonia distillation apparatus [in analysis of lake-waters]. G. Kemmerer and L. T. Hallett (Ind. Eng. Chem., 1927, **19**, 1295—1296).—The ammonia distillation apparatus designed by Parnas and Wagner gives accurate results, but suffers from the disadvantage that its rubber connexions soften in contact with steam and hot alkali. An all-pyrex glass apparatus has been designed which has the advantage that comparatively dry steam is delivered to the actual distillation vessel, special facilities are provided for washing the apparatus between determinations, and no dissolution of the glass by the distillate can be detected. E. Holmes.

Water purification by electro-osmosis. A. S. Behrman (Ind. Eng. Chem., 1927, **19**, 1229—1230).—A series of cells each consisting of three compartments is built up into the shape of a filter-press. The water under treatment passes through the series of middle compartments, while a slow feed of rinse water is maintained in the electrode chambers. The voltage is increased with the decreasing conductivity of the treated water. The power consumption is 75—95 kw.-hrs. per 1000 gallons for complete purification. Soluble sodium and potassium compounds are, of course, removed equally with calcium salts. The process may be combined with a pretreatment by the cheaper lime-soda or zeolite methods. C. Irwin.

Patent.

Compositions for the prevention of boiler scale. H. Karplus (E.P. 255,865, 19.7.26. Switz., 23.7.25).—The compositions containing inorganic substances which can be converted into the colloidal state, *e.g.*, carbon or graphite, are protected by organic colloids which contain organic non-colloids, do not froth, and are stable under boiler conditions. Suitable materials are cellulose waste liquors, alkali humates, or salts of lignic or lignosulphonic acids. W. G. Carey.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DECEMBER 30, 1927.

I.—GENERAL; PLANT; MACHINERY.

PATENTS.

Centrifugal apparatus and process for continuously separating liquids from solids. H. C. Behr (U.S.P. 1,642,662, 13.9.27. Appl., 6.5.25).—The periphery of a distributing member is surrounded by a foraminous separating member mounted within a receiving member. The separating and receiving members rotate respectively at a different speed from, and in phase with, the distributing member. H. Holmes.

Apparatus for separating liquids of different specific gravity. T. Fisher, Assr. to Fisher Engineering Corp. (U.S.P. 1,641,843, 6.9.27. Appl., 14.1.25).—A channel between inner and outer vertical casings is provided with a helical deflector terminating in a vertical portion extending into the lighter liquid zone defined by an enlargement on the inner casing. The liquids are delivered into the channel below the lighter liquid zone at the upper side of the deflector. A discharge pipe for the heavier liquid extends upwards within the inner casing to the lighter liquid zone, and a discharge pipe for the lighter liquid is provided at the upper end of this zone. H. Holmes.

Apparatus for continuous dialysis. O. Faust and H. Vogel (G.P. 441,258, 1.7.24).—A dialysing unit comprises a spiral dialysing tube constructed of cellulose esters, cellulose ethers, or regenerated cellulose, wound around a hollow framework of square, polygonal, circular, or oval cross-section, constructed of wood, ceramic material, metal, or vulcanite, and containing an impermeable core. The units are connected together in a battery in such a manner that the water and dialysing liquid pass through in opposite directions. L. A. Coles.

Liquid and gas contact apparatus. F. H. Wagner, Assr. to Bartlett Hayward Co. (U.S.P. 1,637,975, 2.8.27. Appl., 17.6.25).—A gas-scrubbing tower is divided into chambers by horizontal bubbling plates, each chamber being fitted with a rotating device adapted to produce a transverse spray of liquid. T. S. Wheeler.

Apparatus for removing dust from gases. J. G. Schulz and H. J. M. Loriot (G.P. 441,000, 27.3.24. Conv., 16.5. and 10.7.23).—The gases pass upwards through a chamber containing bundles of chains, so disposed that they are easily removable for cleaning, resting upon a perforated plate. The bottom of the chamber is bowl-shaped, and contains oil or other liquid on which the gas impinges, thus causing it to spray on to the chains. Deposition of the dust is facilitated by varying the cross-section of the chamber in the direction of the gas stream, thereby causing the gas to pass through it with varying velocities. L. A. Coles.

Heat-exchanging device for air or other media. A. Sandberg (E.P. 255,418, 29.4.26. Conv., 18.7.25).—The gas to be heated and that to be cooled flow in parallel chambers or groups of chambers in both of which a number of discs are revolved, half of each disc in each chamber. The partitions between the discs, forming the dividing wall between the gas chambers, are flexibly mounted to allow for expansion and contraction, and make resilient contact with the discs. B. M. Venables.

Viscosimeter. E. H. Peabody, Assr. to Peabody Engineering Corp. (U.S.P. 1,639,057, 16.8.27. Appl., 23.11.22).—A receptacle for a liquid is adapted to be filled in one position and emptied in another position. The movement from the filling to the emptying position brings into operation a device for measuring the duration of the outflow. H. Holmes.

Balance for measuring differences in gravity. "Exploration" Bodenuntersuchungs- u. Verwertungs-Ges.m.b.H. (E.P. 270,754, 7.5.27. Conv., 8.5.26).—The beam of a balance on the Eötvös system is made Z-shaped, the suspending wire being attached to about the middle of the vertical limb and enclosed in the same casing as that limb. The wire may be attached directly to the limb by bending the latter to clear the wire, or it may be in the form of a tube with the wire inside. B. M. Venables.

Apparatus for analysing substances by means of Röntgen or cathode rays. F. Dessauer, Assee. of Metallbank u. Metallurgische Ges. A.-G. (E.P. 252,206, 17.5.26. Ger., 15.5.25. Addn. to E.P. 252,207; B., 1927, 735).—In the apparatus described in the original patent, filters are placed in the path of the rays entering the ionisation chamber by which the characteristic kinks or discontinuities are magnified in relation to the general ionisation–voltage curve. The filters may either effect selective absorption, allowing only the characteristic secondary radiations to be emitted, or they may have an ionisation capacity lower than that of the substance to be examined. B. M. Venables.

Concentrator for liquids. W. Vogelbusch (U.S.P. 1,637,431, 2.8.27. Appl., 7.8.24. Conv., 22.11.23).—See E.P. 196,935; B., 1924, 157.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Water-gas production from coal dust and finely-divided fuels. Gwosdz (Chem.-Ztg., 1927, **51**, 585—586, 606—608).—A critical survey of modern processes designed for producing water-gas from finely-divided

fuels. Proposed methods of continuous water-gas production have hitherto been found impracticable. External heating of the producer presents difficulties, *e.g.*, choice of suitably resistant refractory material, prevention of excessive heat loss in the waste gases. The possibilities of inorganic catalysts are noteworthy, and practical importance attaches to the gasification, by an alternating process, of the fine fuel maintained in suspension. W. T. K. Braunholtz.

Electrolytic corrosion of [gas] mains. A. Bolzinger (Gas J., 1927, **189**, 499—501, 551—553, 628—629, 754—756).—The presence of lead peroxide is definite evidence of electrolytic corrosion of a lead pipe, but its absence does not prove the absence of electrolytic action. Stray currents due to town distribution systems (*e.g.*, lighting or telephones) are normally not serious, but such is not the case with tramway systems. Induced currents may also occur, the system of mains constituting a secondary coil, one of its sides often being parallel to the tram rails, which constitute the inducing coil. Paints and dressings do not generally afford adequate protection against electrolysis, the chief difficulty being their power of absorbing moisture. Other methods of protection (*e.g.*, insulating joints, baffle plates, lowering the potential of the mains, electric drainage of mains) are costly and of doubtful value. It is more logical and effective to suppress stray currents by exacting observance of certain conditions by electricity undertakings. Details are given of French legislation in this connexion. At the same time the gas engineer must exercise a methodical supervision of his mains, including the charting of stray currents and the carrying out of tests periodically at selected points in the system. W. T. K. Braunholtz

Evaluation of transformer oils. J. G. Ford (Ind. Eng. Chem., 1927, **19**, 1165—1171).—Determinations of unsaturated hydrocarbons by absorption in sulphuric acid (*d* 1·84) and oxidation at various temperatures for long periods show that transformer oils freed from unsaturated hydrocarbons are susceptible to oxidation and consequent sludge formation, whereas the presence of unsaturated hydrocarbons considerably retards oxidation, but if present in small amount high acidity and excessive sludge subsequently develop. Oxidation tests with oils containing naphthene and paraffin as their basic compounds, but with the same sulphuric acid absorption values, show that when over 5% of unsaturated hydrocarbons are present the quantity of sludge is independent of the base of the oil. Naphthene hydrocarbons of high mol. wt. are not so sensitive to oxidation as those of low mol. wt., and mixtures of the former class of naphthenes with paraffins give an oil with a high resistance to oxidation and sludging. No general rule can be given concerning the effect of different temperatures on the rate of oxidation of different oils, but high-temperature tests do not correlate with results obtained in actual use; transformer oils should, however, be operated at the lowest possible temperature.
W. G. Carey.

Catalysts and their effects on the oxidation of mineral oils. G. W. Cupit (Refiner Nat. Gasoline Manuf., 1927, **6**, No. 7, 61, 68).—Vanadium pentoxide (except on addition of chromic acid), iron, tin, nickel, aluminium, and cupric oxide are ineffective; reduced copper is effective. The type and weight of residue vary with the time and temperature of reaction. The oils darken and become turbid in sunlight.
Chemical Abstracts.

Solubility of paraffin wax in oil. F. W. Sullivan, jun., W. J. McGill, and A. French (Ind. Eng. Chem., 1927, **19**, 1042—1045).—Paraffin waxes of m.p. 43·9°, 51·7°, and 63·8° were prepared by the repeated fractionation of a commercial wax of m.p. 51·7° from midcontinent crude oil. The solubilities of these waxes in light petroleum and in a number of distillate oils were determined. The cloud temperature was taken as the point of saturation. The results prove that the solubility of paraffin wax increases as the m.p. decreases, that it decreases with increasing viscosity of the solvent, and that these variations are less marked the lower the temperature. C. Irwin.

Heat of solution of paraffin wax. F. W. Sullivan, jun., W. J. McGill, and A. French (Ind. Eng. Chem., 1927, **19**, 1040—1041).—The heat of crystallisation of paraffin wax from the wax distillate out of midcontinent crude oil was redetermined and found to be 72·5 B.Th.U. per lb. A wax-free gas oil was used as solvent.
C. Irwin.

Patents.

Gas analysis apparatus. R. Eisenschitz, Assr. to Bailey Meter Co. (U.S.P. 1,643,155, 20.9.27. Appl., 31.12.26. Ger., 18.1.26).—A pipe conveying the gas is provided with conduit means for withdrawing a gas sample, passing it together with a fluid supporter of combustion through a catalytic analyser, and returning it to the pipe. Means are provided for maintaining a predetermined constant difference between the pressure of the fluid supply and that in the pipe.
H. Holmes.

Gas production. W. W. Kemp (Can. P. 264,747, 12.6.24).—The starting material, *e.g.*, coal, wood, coke, oils, tar, etc., is passed through a closed air-tight retort in which it is subjected to the direct action of a flame produced by the combustion of a mixture of gases and oxygen. A. B. Manning.

Granular material for purifying acetylene. "Hera" Landsberger & Co., and Ver. Chem. Fabr. zu Leopoldshall, Zweigstelle der Kaliwerke Aschersleben (G.P. 442,368, 28.1.25).—The material consists of sintered kieselguhr impregnated with a solution of chromic acid. A. B. Manning.

Low-temperature carbonisation. Metallbank & Metallurgische Ges. A.-G. (G.P. 440,379, 22.7.23).—In the carbonisation of fuels by the circulation through them of a mixture of hot carbonisation gases and a supplementary gas, the former circulates only through the carbonising zone, whilst the latter, before admixture with the former, is passed through the cool zone. In this way a supplementary gas of high water-vapour content is obtained, with consequent improvement in the quality and yield of the tar. A. B. Manning.

Manufacture of light hydrocarbon liquids. W. S. Yard and E. N. Percy (U.S.P. 1,643,401, 27.9.27. Appl., 17.3.24).—Heavy hydrocarbon oils are sprayed upon a

deep bed of incandescent solid fuel, and the evolved vapours are passed over and then through a mass of finely-divided carbonaceous material electrically heated to incandescence. Steam is used to prevent fouling of the mass by cracking products. C. O. Harvey.

Treatment of petroleum hydrocarbons. W. M. Cross, Assr. to Gasoline Products Co. (U.S.P. 1,643,446, 27.9.27. Appl., 13.8.24).—The oil is heated to a cracking temperature, collected in a reaction chamber under such conditions of temperature and pressure as will induce cracking, while maintaining the oil in a liquid state, and is then injected into an enlarged zone maintained at reduced pressure, where the oil stream meets a jet of steam. The volatile products are removed and collected. C. O. Harvey.

Synthesis of hydrocarbons. Thermatomic Carbon Co., Assees. of E. B. Spear (Can. P. 264,324, 28.11.25).—Carbon deposited in a heated retort by the passage of methane through it is decomposed by a current of steam, yielding carbon monoxide and hydrogen, and the gas mixture, after the addition of more hydrogen, is used for the synthesis of hydrocarbons. L. A. Coles.

Purification of hydrocarbons. L. W. Parsons and S. P. Coleman, Assrs. to Standard Development Co. (U.S.P. 1,640,720, 30.8.27. Appl., 10.11.22).—Petroleum is heated with oxide of copper or lead (1—15%) at about 150° for ½ hr., and the product distilled in contact with sodium hydroxide (2—8%). T. S. Wheeler.

Centrifuges, particularly for the purification of oil. Aktiebolaget Separator, Assees. of T. H. Miller (E.P. 271,526, 23.5.27. Conv., 24.5.26).—A centrifugal bowl mounted on a hollow shaft carried by two bearings is kept in position by springs which allow of slight displacements of the rotating bowl by vibration. The oil enters and leaves the bowl *via* inlet and outlet pipes which pass through the bearings and communicate with the hollow shaft and thence with the bowl. By this arrangement entrainment of air and oxidation of the oil are avoided. C. O. Harvey.

Process of treating [cracking] hydrocarbon oils. C. P. Dubbs, Assr. to Universal Oil Products Co. (U.S.P. 1,638,112—3, 9.8.27. Appl., [A], 29.9.19, [B] 17.2.21. Renewed [A, B] 24.3.27).—(A) A continuous stream of oil under pressure is cracked by passage through a heating zone, and enters an enlarged vapour chamber whence the unvaporised residue is withdrawn and kept apart from the incoming charging stock. The condensate formed by passing the vapours through a vapour line and reflux condenser is trapped and returned to the heating zone, the remaining vapours being condensed and collected. (B) The oil is cracked in a horizontal cylindrical still and the vapours pass to a dephlegmator, the reflux condensate being returned to the base of the still where the temperature is highest. The precipitated carbon is collected out of contact with the lower heated portion of the still. C. O. Harvey.

Conversion [cracking] of hydrocarbon oil. L. C. Huff, Assr. to Universal Oil Products Co. (U.S.P. 1,638,735, 9.8.27. Appl., 18.11.25).—The oil flows in a continuous restricted stream into a furnace-heated coil, and thence the vapours and unvaporised oil pass to an enlarged reaction zone where conversion occurs. The mixture now passes to a zone maintained at a lower pressure wherein the more volatile portions vaporise and are separated from the residual oil. Condensation of the vapours takes place in reflux condensers cooled by means of charging oil, which is thereby preheated. C. O. Harvey.

[Cracking] treatment of heavy hydrocarbons. A. D. Smith and J. Perl (U.S.P. 1,640,223, 23.8.27. Appl., 28.1.22).—The oil in circulation is partially cracked by heating under pressure whereby gaseous and semi-liquid polymerised hydrocarbons are formed, the latter remaining in solution in the hot residual oil, which then passes to a cooler zone, where precipitation occurs. Thence the residual oil passes back to a second heating zone, where it is mixed with a further quantity of the original oil stock. C. O. Harvey.

Apparatus for cracking hydrocarbons. G. Egloff and J. C. Alther, Assrs. to Universal Oil Products Co. (U.S.P. 1,638,116, 9.8.27. Appl., 23.12.20. Renewed 24.3.27).—The oil, cracked in a heated coil, passes to an expansion chamber connected with a reflux condenser cooled by means of charging stock. The residual oil from the expansion chamber is used to preheat incoming charging stock, which passes, along with the heated charging stock and condensate from the reflux condenser, to the cracking coil. C. O. Harvey.

Apparatus for cracking hydrocarbons. J. E. Bell (L. R. Bell, extrix.) and E. W. Isom, Assrs. to Sinclair Refining Co. (U.S.P. 1,641,941, 6.9.27. Appl., 25.7.25).—A horizontal cylindrical still member, divided into communicating compartments, is situated above a furnace fitted with flues and adapted for re-circulation of the flue gases. Heating pipes pass downwards into the furnace from each compartment of the still, those connected to the supply compartment being nearest to the fire box. C. O. Harvey.

Apparatus for cracking mineral oils and the like. Werschen-Weissenfelser Braunkohlen A.-G., and A. Fürth (G.P. 439,520, 18.1.23. Addn. to G.P. 439,010; B., 1927, 695).—The catalyst comprises a fine-grained material which is heated directly by passing a current through it. To ensure efficient insulation from the conducting walls of the autoclave, the current is led in by a cooled tube, the cold end of which is provided with a stuffing-box or other arrangement to prevent leakage of the high-pressure gases. A. R. Powell.

[Demulsifying] treatment of hydrocarbons. A. B. Way, Assr. to L. A. Way (U.S.P. 1,638,021—2, 9.8.27. Appl., 19.3.24).—Emulsions of hydrocarbon oils are broken and the fluidity of the oils is increased by treatment for about 10 hrs. at below 95° with 0·02% of a finely divided mixture of naphthalene (95·5%) and nitrobenzene (4·5%). T. S. Wheeler.

Dehydration of oils. S. Born, H. E. Bonnette, and J. C. Walker, Assrs. to Empire Gasoline Co. (U.S.P. 1,639,325, 16.8.27. Appl., 20.4.20).—Water is separated from petroleum, treated with an alkali hydroxide in an amount less than that required substantially to affect its hardness, and the alkaline solution then mixed with the petroleum. C. O. Harvey.

Preparation of an adsorbent for oils. W. D. RIAL and E. W. GARD (U.S.P. 1,639,274, 16.8.27. Appl., 26.4.26. Cf. U.S.P. 1,634,514 ; B., 1927, 771).—Clay is agitated with a mixture of petroleum hydrocarbons and aqueous alkali, separated from the excess of hydrocarbons, and recovered. C. O. HARVEY.

Oil-refining apparatus. H. A. W. HOWCOTT (U.S.P. 1,641,852, 6.9.27. Appl., 7.2.24).—The oil passes through a number of horizontal preheaters rigidly attached to a vertical cracking chamber and communicating therewith beneath the surface of the oil. A rotating horizontal shaft passing through the apparatus carries paddles for agitating the oil in the preheaters and propellors on either side of each paddle, to force the oil towards the paddle. The shaft is also provided with cranks and means attached thereto for agitating the oil in the cracking chamber, from which the vapours are withdrawn, and in which a predetermined pressure is maintained. C. O. HARVEY.

Refining of hydrocarbon oils. E. B. PHILLIPS and J. G. STAFFORD, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,639,531, 16.8.27. Appl., 22.10.24).—Sulphuric acid-refined oil, treated with an alkali, is passed down a reflux tower in direct contact with upcoming vapours from a still situated below the tower. C. O. HARVEY.

Recovering styrene [from carburetted water-gas drip-oil]. R. L. BROWN (U.S.P. 1,640,975, 30.8.27. Appl., 10.11.22).—Styrene is recovered from carburetted water-gas drip-oil by a process involving fractionation followed by conversion of the styrene into a relatively non-volatile form from which other compounds are separated by distillation. C. O. HARVEY.

Process for recovering vanadium from petroleum hydrocarbons. A. OBERLE, Assr. to T. E. SCOFIELD (U.S.P. 1,638,057, 9.8.27. Appl., 8.9.25).—The exhaust gases of Diesel engines working on heavy oils containing vanadium are passed through activated charcoal to separate vanadium oxide. T. S. WHEELER.

Manufacture of a mineral oil derivative. G. ALLEMAN, Assr. to SUN OIL Co. (U.S.P. 1,637,510, 2.8.27. Appl., 6.2.24).—Alkali sludge obtained in refining petroleum is heated with water under a pressure of 4 atm. for 2 hrs., the solution obtained being concentrated to separate soaps and resins. This mixture is treated in aqueous solution with a metallic salt, *e.g.*, calcium chloride, to give a precipitate of value as an adhesive, filler, etc. T. S. WHEELER.

Production of a carbon catalyst. C. B. JACOBS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,623,598, 5.4.27. Appl., 27.11.20. Cf. U.S.P. 1,462,752 ; B., 1923, 975 A).—Black liquor from the soda-cellulose process is evaporated, and the residue incinerated at 200—350°. After washing out soluble matter with water, a highly active carbon remains. T. S. WHEELER.

V.—FIBRES ; TEXTILES ; CELLULOSE ; PAPER.

Oxidation of alkali-cellulose by gaseous oxygen. W. WELTZIEN and G. ZUM TOBEL (Ber., 1927, **60**, 2024—2032 ; cf. B., 1926, 911).—Cotton wool or hydrated cellulose is immersed in sodium hydroxide solutions of different concentrations ; the products are squeezed and exposed to the action of oxygen at 60°. The sodium hydroxide is thus present partly in combination with the cellulose as established by Vieweg, and partly adherent to the fibre (swelling hydroxide). With increasing concentration of alkali in the bath up to 30%, the absorption of oxygen increases considerably, and attains remarkably high values ; reaction is complete within about 15 days. With higher concentrations the absorption is smaller, but is less rapidly completed. If the primary oxidation consists in the conversion of a carbonyl group into a carboxyl group which is neutralised by the alkali or in the complete combustion of the cellulose, the absorption of 1 atom of oxygen is causative of the neutralisation of 1 mol. of sodium hydroxide, and the established, practical cessation of the reaction when the greater part of the alkali has been neutralised is readily understood. The rate of absorption of oxygen depends on the concentration of the alkali, and attains a maximum of 23·8%. With hydrated cellulose, owing to the presence of greater amounts of "swelling alkali," the rate of absorption of oxygen and the amount of oxygen absorbed are higher than with cotton wool. The absorption is also influenced by the extent to which the material is squeezed and the density of its packing into the apparatus. Acidification of the resulting solution causes the evolution of large amounts of carbon dioxide and the separation of alkali-soluble cellulose, which shows only slightly pronounced signs of oxidation. It appears that the reaction proceeds in such a manner that greatly oxidised fission products are comparatively rapidly eliminated. In an atmosphere of nitrogen, the alkali solubility increases gradually in the course of a few days, and subsequently remains nearly constant. The reaction certainly differs fundamentally from that which occurs in oxygen, and the diminution of alkalinity in the solution observed under the latter conditions is almost entirely to be ascribed to the action of oxygen. H. WREN.

Inorganic solvents for cellulose. H. E. WENNSTROM (Rayon, 1927, **4**, No. 11, 20—22).—The peptising action of saline solutions on cellulose is attributed to the combination of the hydroxyl groups of the cellulose molecule with molecules of anhydrous salts, forming compounds in which the cellulose is substituted for water of crystallisation. CHEMICAL ABSTRACTS.

Viscose silk. IV. Analysis of viscose. I. FUKUSHIMA, Y. TAKAMATSU, and I. WATANABE (J. Cellulose Inst., Tokyo, 1927, **3**, 214—219).—The analytical procedure for determining the distribution of the alkali in crude viscose is modified to include alkali combined as trithiocarbonate. D. J. NORMAN.

Mechanism of the loading of paper. K. ATSUKI and M. NAKAMURA (J. Cellulose Inst., Tokyo, 1927, **3**, 49—60).—The amount of china clay fixed by sulphite pulp from a neutral suspension in water under standard conditions increases with increasing concentration of the clay until the latter reaches 3—4%, when a constant maximum value is attained. The addition of starch paste or acidified viscose solution to such a suspension greatly increases the amount of clay fixed, no increase being obtained, however, if the cellulose is not regenerated

from the viscose. Glue acts as a protective colloid to the clay particles and prevents enhanced fixation. The increase which results from the addition of aluminium sulphate is attributed to the action of the acid liberated from the salt by hydrolysis. For different pulps the amount of clay fixed is determined by their physical nature and condition, prolonged beating of the pulp causing an increase, whilst prolonged agitation of the clay and pulp mixture causes a decrease. It is concluded that occlusion of the clay by the fibre is the cause of the fixation and that adsorption plays a minor part. The hydrogen-ion concentration of the medium is of importance, since the clay particles tend to become aggregated by acid and peptised by alkali, so that greater occlusion takes place in slightly acid suspensions.

B. P. Ridge.

Colloid chemical investigation of sulphite lye. III. Fractions obtained by electro-dialysis. M. Samec and I. Ribarič (Koll.-Chem. Beih., 1927, **24**, 157—180; cf. Samec and Rebek, *ibid.*, 1923, **19**, 106).—Dialysed sulphite lye, if concentrated by electro-dialysis and then subjected to further electro-dialysis until the conductivity of the cathode cell liquid reaches a constant value, is separated into two portions, one of which appears in the middle cell (the "colloidal residue"), the other in the anode cell. The anode cell product is concentrated *in vacuo* at 45°, and, on repeated electro-dialysis, the greater part of the organic matter remains in the middle cell (the "anode colloid"), whilst the free sulphuric acid and the constituents of lower mol. wt. appear in the anodic compartment. Investigation of the colloidal residue, by fractional precipitation of the barium and β-naphthylamine salts, showed it to be a well-defined ligninsulphonic acid. By determination of the sulphur content, the titratable acidity, the methoxyl content, and the acetyl content, the values 551, 549, 219, and 1096, respectively, were obtained for the equivalent weight, and the value 1100 for the minimum mol. wt. Cryoscopic measurements lead to the value $M = 1136$. From measurements of the alteration of the equivalent conductivity of the free acid and of the sodium salt with increasing dilution, and the application of the valency rule, it is concluded that the acid is dibasic. The mol. wt. of the "anode colloid" is smaller than that of the ligninsulphonic acid, the methoxyl content is less, and the number of acid groups greater. The individual acid groups are ionised to different extents. Assuming these to be carboxyl groups, a scheme is suggested to show the relation between the ligninsulphonic acid and the ligninsulphocarboxylic acid.

L. L. Bircumshaw.

Patents.

Production from viscose of artificial formations suitable for the textile industry. Borvisk Synd., Ltd., Assees. of B. Borzykowski (E.P. 273,647, 27.11.26. Ger., 2.7.26).—Artificial filaments with a matt appearance are obtained from ordinary sodium viscose by adding to the spinning solution suitable quantities, *e.g.*, 5% on the weight of cellulose, of oils, fats, soaps, emulsified mineral oils, hydrogenated aromatic hydrocarbons such as tetralin.

D. J. Norman.

Manufacture of hollow articles from cellulose or its derivatives. Wolff & Co., E. Czapek, and F. Bauer (E.P. 276,206, 27.11.26).—In the manufacture of hollow articles, *e.g.*, bottle caps, from viscose solution or other solution containing cellulose or a cellulose derivative, special effects may be obtained by superimposing two or more layers of viscose on the mould before coagulation. In this way a thin adherent film of colourless or suitably dyed viscose may be superimposed on an opaque viscose or one containing an iridescent loading material to give a glossy finish and a metallic appearance.

D. J. Norman.

Production of pulp having a high percentage of resistant cellulose. G. A. Richter, Assr. to Brown Co. (U.S.P. 1,640,853, 30.8.27. Appl., 27.5.24).—Wood is first digested under pressure with a hot solution of sulphurous acid containing sodium sulphite, the liquor then made alkaline, and the digestion continued.

B. P. Ridge.

[Treatment of] wood-pulp paste etc. Ramar Syndicate, Inc. (F.P. 615,809, 8.5.26).—For the treatment of the vegetable material comparatively dilute solutions of salts (sulphates of sodium, potassium, magnesium, iron, etc., or mixtures of these) are used in order to loosen the intercellular binding material without dissolving the lignin. If necessary, a mixture with the soluble salts of chromium, cobalt, nickel, or zinc is used.

B. P. Ridge.

Artificial product from absorbent material. W. Heuer and A. Lang (G.P. 440,813, 21.11.23).—The material (paper, paper or textile waste, etc.), after treatment with water-glass and talc in the form of layers or paste, is dried, then steeped in a bath of hydrochloric acid, and immediately washed with water. The products are resistant to weather etc.

B. P. Ridge.

Process of elaborating corn stalks into pulp for paper-making etc. O. R. Sweeney (U.S.P. 1,639,152, 16.8.27. Appl., 15.11.26).—The material is cooked with sodium hydroxide, lime, sulphite, etc., the mass submitted to bacterial action to promote partial disintegration of the stalks, and finally treated in a mechanical beater.

B. P. Ridge.

Recovery of pulp from printed waste paper. E. T. McGregor (U.S.P. 1,640,612, 30.8.27. Appl., 21.6.26).—The paper is treated with a solution of trisodium phosphate, which is kept cool, then pulped, beaten, unknotted, and finally washed with cold water.

B. P. Ridge.

Improving the spinning properties of cellulose solutions. Chem. Fabr. Pott & Co. (F.P. 617,600, 16.6.26. Ger., 17.6. and 15.12.25).—Alkylated naphthalenesulphonic acids, or their salts or derivatives, produced by condensing naphthalenesulphonic acids with *iso*propyl or *sec.*-butyl alcohol, are added to the solution and/or the precipitation bath.

L. A. Coles.

Treatment of acetic acid [mother] liquor [from cellulose acetate manufacture]. Soc. Chim. des Usines du Rhône (E.P. 266,684, 19.1.27. Fr., 26.2.26).—The precipitation of dissolved cellulose during the recovery of acetic acid is prevented by heating the dilute acid for, *e.g.*, 6 hrs. at 65° or 2 hrs. at 90°, with a small quantity of an acid which is capable of hydrolysing the dissolved cellulose to water-soluble products

e.g., 10 c.c. of sulphuric acid (*d* 1·53) per litre of 27% acetic acid. The sulphuric acid is subsequently neutralised with sodium carbonate. D. J. Norman.

Recovery of ammonia from cuprammonium precipitation baths. "Cupram" A.-G. (Swiss P. 117,969, 26.3.25. Ger., 6.5.24).—Magnesium ammonium phosphate is precipitated by the addition of magnesium hydrogen phosphate to the bath. L. A. Coles.

Manufacture of cellulose compounds. L. Lilienfeld (U.S.P. 1,642,587, 13.9.27. Appl., 23.7.24. Conv., 4.4.24).—See E.P. 231,800; B., 1925, 913.

Manufacture of artificial materials. L. Lilienfeld (U.S.P. 1,642,588, 13.9.27. Appl., 23.7.24. Conv., 4.4.24).—See E.P. 231,802; B., 1925, 913.

Production of artificial silk [by the dry-spinning process] and apparatus therefor. Courtaulds, Ltd., F. Shedden, A. E. Delph, and N. G. Baguley (E.P. 275,042, 4.10.26).

Carbon catalyst (U.S.P. 1,623,598).—See II.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing of neps. L. G. Lawrie (J. Soc. Dyers and Col., 1927, **43**, 294—295).—The affinity for direct dyes of neps consisting of dead cotton hairs (cf. Slater, B., 1927, 551) frequently present in cotton fabrics is increased so as to equal that of normal cotton by mercerisation with strong caustic soda, with or without tension, this result being contrary to the conclusions of Clegg and Harland (B., 1923, 709 A). Many sulphur, basic, and anthraquinone vat dyes dye neps satisfactorily, but indigo and some indigoid vat dyes are resisted by neps. A list of twenty-four direct dyes which are most suitable for dyeing neps is given. About 50% of dyes which coloured viscose silk did not colour neps. A. J. Hall.

Method for detecting complete development of Indigosols and Soledon colours on wool. H. R. Hirst and P. E. King (J. Soc. Dyers and Col., 1927, **43**, 266—267).—Aqueous solutions and dyeings on wool of Soledon and particularly Indigosol dyes show a strong fluorescence when exposed to ultra-violet light, this fluorescence being destroyed by complete oxidation to the normal vat dye. The presence of an incompletely developed (oxidised) Indigosol dye on wool may thus be readily detected. Wool dyed with Indigosol O and incompletely developed is fluorescent in ultra-violet light, but when subsequently exposed to light from a Fadeometer the shade develops progressively and is accompanied by a decrease in fluorescence. A. J. Hall.

Patents.

Production of printed and dyed effects on artificial silk fabrics. Calico Printers' Assoc., Ltd., J. R. Whinfield, and C. Levin (E.P. 273,011, 26.3. and 25.5.26).—A cellulose acetate fabric is printed with caustic alkali (*e.g.*, 40% sodium hydroxide, suitably thickened), the alkali is washed off, and the fabric is treated with a delustring agent (*e.g.*, boiling water, aqueous phenols or naphthols, aqueous aniline or pyridine, formic, acetic or lactic acids, acetone, or furfuraldehyde), leaving a lustrous pattern on a matt ground. The pattern may be dyed with direct dyes, the matt ground with dyes for acetate silk, or either may be left colourless. C. Hollins.

Dyeing cellulose acetates. I. G. Farbenind. A.-G., Assees. of E. Müller and A. Schaeffer (G.P. 438,378, 1.12.22).—Ammonium salts (excluding the thiocyanate) are added to the dye-bath, whereby good dyeings are obtained, without acidification, with basic, acid, and some substantive dyes. No claim is made to dyeing with salts of basic dyes with the aid of ammonia. C. Hollins.

Dyeing acetate silk. I. G. Farbenind. A.-G., Assees. of A. Aurednicek, E. Keiner, and R. Krech (G.P. 439,111, 21.12.24).—Acetate silk is dyed in an aqueous bath with insoluble or sparingly soluble dyes or pigments (excluding colour bases), with the addition of soap and organic compounds (amyl alcohol, glycerol, glycerol monochlorohydrin, *cyclo*hexanol, phenol, *p*-toluenesulphon-amide or -anilide, etc.) which form easily dissociated salt-like alkali derivatives. An improved exhaust is claimed. C. Hollins.

Dyeing cellulose acetate. G. Lacroix (F.P. 603,123, 12.9.25. Conv., 2.6.25).—The dye (*e.g.*, benzeneazophenylmethylpyrazolone, hexahydroxyanthraquinone, Algol Brilliant Violet 2B) is dissolved in a chlorinated or hydrogenated hydrocarbon (*e.g.*, trichloroethylene, tetra- and penta-chloroethane, chlorobenzene, chlorotoluenes, turpentine, tetralin, decalin, etc., or mixtures of these), with addition of soap, and poured into a boiling soap solution. The mixture, after sieving, constitutes the dye-bath. C. Hollins.

Dyeing and printing cellulose acetate silks. A.-G. für Anilin-Fabr. (F.P. 605,127, 29.10.25. Conv., 3.12.24).—Cellulose acetate silk is treated in presence of ammonia and an oxidising agent with arylamines or their derivatives, or with mixtures of amines and phenols suitable for production of indamines or indophenols. Treatment with *pp'*-dihydroxydiphenylamine, ammonia, and Turkey-red oil at 40° gives a brownish-red on acetate silk; 3 : 5-dichloro-4-amino-4'-hydroxydiphenylamine, a bluish-red; 4-dimethylamino-4'-hydroxydiphenylamine, a blue; 4-amino-4'-hydroxydiphenylamine, a violet. By the use of *p*-phenylenediamine, ammonia, and hydrogen peroxide at 50—60° a brown shade is obtained, which is deepened by after-treatment with copper sulphate and alkali dichromate in acetic acid. The bronze-brown obtained with *p*-aminodiphenylamine, ammonia, and ferricyanide becomes by similar treatment violet-brown. An alkaline solution of *p*-hydroxyphenyl-β-naphthylamine, ferricyanide, and ammonium chloride give a reddish-orange; similarly *pp'*-dihydroxydiphenylamine gives a full red, a mixture of *p*-phenylenediamine and phenol a violet, *p*-phenylenediamine and dimethylaniline a deep violet, and *p*-aminophenol and dimethylaniline a blue. C. Hollins.

Rendering cellulose acetate silk etc. resistant to hot or boiling aqueous liquors. Silver Springs Bleaching & Dyeing Co., Ltd., and A. J. Hall (E.P. 277,089, 8.6.26).—Cellulose acetate filaments, in fully set and finished condition, are stretched beyond their elastic limit (*e.g.*, 15—25% elongation), with or without a previous treatment with a swelling agent (acetone, alcohol, formaldehyde, glycerol). The fibre can then be

boiled with water for 1 hr. without curling, losing lustre, or becoming woolly. C. Hollins.

Manufacture of aqueous solutions or emulsions of solvents or other liquids or solids insoluble in water. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 277,277, 10.3.26).—Aqueous emulsions or solutions of insoluble liquids or solids, particularly for the treatment of fibrous materials, are made with the help of an emulsifying agent consisting of a sulphonated polynuclear hydrocarbon (naphthalene) containing a side-chain of 5 or more carbon atoms. Naphthalene-β-sulphonic acid, *e.g.*, may be condensed in sulphuric acid with amyl or hexyl alcohol and the product used as an emulsifying agent. C. Hollins.

Treatment of vegetable fibres with alkalis, or with oxidising or reducing agents. M. Bergmann, E. Immendörfer, and H. Löwe (G.P. 440,996, 29.3.23. Cf. G.P. 426,624; B., 1926, 785).—Solutions used in processes such as washing, bleaching, Aniline Black dyeing, discharging, reserving, boiling with alkalis, etc. contain natural or artificial tanning agents in quantity insufficient for the complete reduction of any oxidising agents present. L. A. Coles.

Fireproofing organic fibrous material. H. Stelling (G.P. 439,765, 7.1.25. Addn. to G.P. 429,918; B., 1926, 858).—Silicon halides other than the chloride, oxyhalides, hydrides, or halogen hydrides, or organic silicon compounds, are used instead of silicon chloride in the process described previously. L. A. Coles.

Production of fast-coloured discharges on fast dyeings. W. Cotton, Assr. to Grasselli Dyestuff Corp. (U.S.P. 1,638,475, 9.8.27. Appl., 16.1.26. Ger., 19.1.25).—See E.P. 246,183; B., 1927, 363.

Dyeing and printing with phenylamine black. G. Aris (U.S.P. 1,643,233, 20.9.27. Appl., 16.2.22. Spain, 1.3.21).—See E.P. 176,343; B., 1923, 651 A.

Manufacture of stable derivatives of vat dyestuffs. A. Wolfram, Assr. to Durand & Huguenin Soc. Anon. (U.S.P. 1,639,206, 16.8.27. Appl., 11.3.27. Conv., 17.3.26).—See E.P. 267,952; B., 1927, 772.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Patents.

Apparatus for the production of ammonium sulphate. Bayerische Stickstoff-Werke A.-G. (G.P. 441,260, 24.8.22).—Ammoniacal gases and vapours are passed into sulphuric acid solutions on the counter-current principle in an apparatus comprising a saturation vessel of small cross-section connected by means of a short tube with a second vessel of broader cross-section. This serves as a crystallising chamber, and is connected with a third vessel which acts as an equaliser, and, by its arrangement, provides additional acid and liquid to make up for any variation in the circulating solution. Liquid is withdrawn periodically from the third vessel, heated, and passed into the saturator. The process effects almost complete utilisation of the heat of neutralisation of the ammonia. A. R. Powell.

Manufacture of precipitated calcium carbonate. B. G. Wood, Assr. to Ivanpah Lime & Chemical Co. (U.S.P. 1,641,563, 6.9.27. Appl., 8.9.25).—Intimate and finely-divided contact is made between an aqueous suspension of calcium hydroxide and carbon dioxide at about 35°, the resulting finely-divided calcium carbonate being then separated and dried. W. G. Carey.

Production of lead suboxide. L. Gutersohn (G.P. 440,978, 11.12.23).—A mixture of lead monoxide with the equivalent weight of an organic acid or ester is heated rapidly and kept for a short time, *e.g.*, spread out in thin layers, at 330° in the absence of air. L. A. Coles.

Production of lead acetate. W. Stern (G.P. 441,112, 9.11.24).—Lead is treated with acetic acid and air at 60—90°, the temperature being maintained by the heat of reaction or by external heating. L. A. Coles.

Production of voluminous bismuth silicate. J. Zeltner (G.P. 440,623, 5.3.25. Addn. to G.P. 433,526; B., 1927, 218).—Bismuth silicate precipitated as described in the prior patent is washed with organic liquids miscible with water, before drying, and the liquid is removed as completely as possible. L. A. Coles.

Production of finely-divided cuprous oxide. I. G. Farbenind. A.-G., Assees. of G. Jantsch and F. Bencker (G.P. 440,755, 23.3.23).—The oxide is formed by the electrolysis of hot alkali or alkaline-earth halide solutions using copper anodes, alkali or alkaline-earth hydroxides being added to the electrolyte during the process. L. A. Coles.

Method and apparatus for condensing aluminium chloride. G. L. Prichard and H. Henderson, Assrs. to Gulf Refining Co. (U.S.P. 1,641,503, 6.9.27. Appl., 3.2.23).—A gaseous mixture containing aluminium chloride vapour is passed through a tubular heat-radiating chamber the interior walls of which are scoured to produce the chloride in a finely-divided state and to prevent accumulation of solid chloride thereon. W. G. Carey.

Production of magnesia from ores containing magnesium oxide or carbonate, such as dolomite. F. van den Bergh (F.P. 616,644, 25.5.26).—The ore is treated with sodium bisulphate to obtain magnesium sulphate, and this is converted into the oxide by treatment with sodium carbonate and subsequent calcination of the magnesium carbonate obtained thereby. L. A. Coles.

Production of alkali silicates. L. Hackspill and J. Salomon (F.P. 617,739, 17.6.26).—Alkali chlorides are heated with silica at 1000° *in vacuo*, or in a current of gas, so that hydrogen chloride and chlorine are removed. L. A. Coles.

Production of crude alkaline-earth sulphides. H. Schulze (G.P. 442,024, 15.7.25).—A mixture of lumpy or powdered barytes, celestine, or gypsum with the theoretical quantity of coal or less, is heated in an electric arc furnace under such conditions that the material sinters superficially and forms a protective coating around the remainder. L. A. Coles.

Production of barium carbonate suitable for the manufacture of pure barium oxide. Rhenania-Kunheim Ver. Chem. Fabr. A.-G. (G.P. 441,736, 18.6.24).—Commercial barium hydroxide is treated with alkali carbonates. L. A. Coles.

Production of compact aluminium hydroxide. J. D. Riedel A.-G. (G.P. 442,255, 25.2.20).—Solid aluminium salts are treated with ammonia solution saturated with ammonium sulphate. L. A. Coles.

Alumina coagulant. H. McC. Spencer, Assr. to Seydel Chem. Co. (U.S.P. 1,643,962, 4.10.27. Appl., 16.4.24).—A coagulant for clarifying aqueous liquids is made by adding to a concentrated solution containing alumina sufficient sulphur dioxide to peptise the alumina. W. G. Carey.

IX.—BUILDING MATERIALS.

Patents.

Calcining Portland cement in rotating kilns. H., R., and I. Dormann (G.P. 440,747, 5.12.24).—The calcining zone is provided with an attachment containing channels for conveying the material under treatment, and with axial or approximately axial conduits through which a portion of the air for combustion is drawn into the flame. L. A. Coles.

Production of cement and other binding agents. Soc. des Ciments Franç. et Bureau d'Organisation Économique (F.P. 612,391, 26.6.25).—The raw material is conveyed in small wagons through a tunnel kiln in which it passes successively through a drying and causticising zone, a clinkering zone, and a cooling zone. L. A. Coles.

Production of cement and mortar impervious to water. Ciment Portland Artificiel de Pont-à-Vendin, Anc. Établ. E. Cambier (F.P. 612,129, 22.6.25).—Material such as cement or its constituents, sand, slag, ashes, shale, clay, gypsum, coke, bauxite, or pyrolusite, or mixtures of these, ground to pass 10,000-mesh, is added to Portland cement or to unground clinker. Solutions containing alkali hydroxides, carbonates, or chlorides, magnesium chloride and lime, mineral acids, oils, fats, bitumen, or albumins or their decomposition products, such as lysalbic acid, may also be added. L. A. Coles.

Production of unsintered hydraulic cement. Rekord-Zement-Ind. G.m.b.H., and O. Tetens (G.P. 440,795, 4.3.23).—An intimate mixture of clay with quick, dry, or slaked lime is calcined, using oil shale as fuel, and the product is ground to cement. Burnt shale may be added to the original mixture, and limestone or lime marl at any stage of the process. L. A. Coles.

Preparation of a plastering mortar. C. A. Kapferer (F.P. 612,932, 18.3.26).—Prepared mortar is treated with solid or dissolved acidic substances, such as calcium hydrogen phosphate, ammonium hydrogen carbonate, calcium hydrogen carbonate, or gelatinous silica, or with insoluble unoxidisable organic substances, such as wax or mineral oils, preferably together with glycerides. The product is waterproof but allows permeation of air. A. R. Powell.

Process and apparatus for burning clay products. J. A. Reams, Assrs. to Fulton Brick Works (U.S.P. 1,639,271, 16.8.27. Appl., 2.12.26).—A kiln furnace having a fuel grate, a shovel-stoking door, and a rear flame exit leading to the kiln is provided with entrances for air and fuel situated in the top of the furnace and having closure devices. C. O. Harvey.

Fire- and weather-proofing wood. H. Suida and H. Salvaterra (Austr. P. 105,361, 27.12.22).—The wood is first impregnated with an aqueous solution of a metallic salt, *e.g.*, the double salt of calcium chloride and calcium acetate, sodium silicate, a magnesium salt, etc., and then again impregnated with a difficultly or non-inflammable chlorinated naphthalene, the second impregnation preventing the subsequent washing-out of the fire-proofing inorganic salt. For soft woods the first impregnation requires only a simple immersion in or coating with the liquid and then drying at 15°, but for hard woods a pressure-vacuum process is necessary. The second impregnation is carried out by immersion either in an easily melting, wax-like chlorinated naphthalene at higher temperatures, or in a solution of the same material in tar or petroleum hydrocarbons or chlorinated hydrocarbons at 15°, or by using a liquid chlorinated naphthalene at 15° in one of the known technical impregnating processes. A. B. Manning.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Oxygen in iron and steel. P. Oberhoffer, H. J. Schiffler, and W. Hessenbruch (Stahl u. Eisen, 1927, 47, 1540—1543).—The solubility of oxygen (as ferrous oxide) in iron appears to be of the order of 0·05—0·06%; its presence is marked by a more electropositive potential and by a more pronounced "primary structure" when the metal is etched with alcoholic cupric chloride. Silicon and more especially manganese reduce the amount of oxygen (as oxide) retained in solid solution, but neither effects complete removal of dissolved oxide. A highly oxidised low-carbon steel is not sufficiently deoxidised with the usual amount (0·1%) of aluminium, and is characterised by more or less red-shortness which can be removed only by the addition of at least 0·84% Al. With 2% Al a hypereutectoidal steel forges well, but gives low impact resistance values due to the presence of inclusions of thin films of alumina. The simultaneous presence of 0·03% O and 0·014—0·020% S is sufficient to cause distinct red-shortness. The presence of dissolved oxide in steel hinders the cementation process, but an excess of aluminium above 1% has a much greater retarding action on the diffusion of the hypereutectoidal zone away from the surface of the metal. Prolonged over-heating of steel containing many oxide inclusions results in the formation of groups of nitride needles around the inclusions, and decarburisation tests at 1050° showed a more rapid loss of carbon than in normal steels. A. R. Powell.

Deoxidation of iron with manganese. P. Oberhoffer and H. Schenck (Stahl u. Eisen, 1927, 47, 1526—1536).—The work of earlier investigators concerning the reaction $FeO + Mn \rightleftharpoons MnO + Fe$ is critically reviewed. As there is no known method of determining the separate proportions of ferrous and manganous oxides present in iron, the equilibrium constant can be determined only indirectly in the following way. On addition of manganese to iron containing oxygen part of the manganese is oxidised and goes into the slag and part remains alloyed with the iron; the part going into the slag takes a certain

amount of ferrous oxide with it, whilst small quantities of both oxides remain in the metal phase. Designating the constituents of the slag with symbols in round brackets and the constituents of the metal with symbols in square brackets, then, if L is the partition coefficient and $K' = [O]_{Fe}[Mn]/[O]_{Mn}$, $K'/[Mn] + 1 = \Sigma[O]L/(MnO)$. In this equation K′ and L are the two unknowns, and these have been determined from the analysis of several melts at 1600°. The results obtained indicate that the equilibrium constant is a function not only of the temperature, but also of the ferrous oxide content of the metal, and that the analytical figures obtained for the oxygen content of the metal are lower than the true values when the manganese content is relatively high. With up to 0·4% Mn in the iron the ratio (MnO)/(FeO) is a linear function of [Mn].

A. R. Powell.

Influence of oxygen on the properties of steel. W. Eilender and W. Oertel (Stahl u. Eisen, 1927, **47**, 1558—1561).—With increase of oxygen content to about 0·03%, mild steel becomes more and more brittle and distinctly red-short with 0·11% O, whilst 0·05% O in knife steel results in a poor polish being obtained owing to the formation of small pits on the surface in places where the minute slag inclusions have been removed by the polishing operation. More than 0·035% O in tungsten tool-steel decreases the effective cutting life, and more than 0·05% O prevents the cold-working of the metal. The permeability and wattage losses of transformer iron are at a minimum when the carbon and oxygen contents are equal. The tendency to "blue brittleness" in ingot steel is increased by the presence of oxygen—no brittleness occurring after a low-temperature anneal if less than 0·03% O is present and very marked brittleness with 0·06%. In view of these results the necessity of controlling the oxygen content of the steel bath during its treatment in the furnace is emphasised. A. R. Powell.

Normal and abnormal steels. S. Epstein and H. S. Rawdon (Trans. Amer. Soc. Steel Treat., 1927, **12**, 337—375).—The abnormality of some case-hardened steels is shown to relate both to grain size and thickness of the carburised layer. The small irregular grain size may be removed by normalising, but the structural abnormality is not affected. With drastic quenching of normal and abnormal steels the degree of hardening is essentially the same, but with less drastic quenching of abnormal steel shows a greater tendency to soft spots. The presence of dissolved gases in the quenching medium has a pronounced effect in the formation of soft spots. No connexion was found between the gas content of the steels themselves and the occurrence of abnormality. The abnormal condition is considered to have its origin in the deoxidation process during the making of the steel. Abnormal steel was found to have a higher aluminium content. Addition of aluminium or ferrovanadium in the mould produced abnormality. T. H. Burnham.

Normality of steel. J. D. Gat (Trans. Amer. Soc. Steel Treat., 1927, **12**, 376—412).—Abnormality of case-hardened steel is shown to be connected with the presence of iron–carbon–oxygen eutectoid films or layers in the cementite mesh surrounding the crystals in the hyper-eutectoid zone. These layers hinder heat-transfer on quenching and decrease the rate of passage through the critical temperature to the extent that troostite is formed instead of martensite. T. H. Burnham.

Properties and heating treatment of cast iron. F. B. Coyle (Trans. Amer. Soc. Steel Treat., 1927, **12**, 446—465).—The results of over 3000 tensile tests are plotted on a Maurer constitutional diagram for cast iron, the maxima all falling within the pearlitic zone. This type of iron showed a true elastic limit at 3·6 tons in tension and 20 tons in compression. The tensile strength of a Diesel engine iron was well maintained in tests up to 482°. The resistance to wear of cast iron was found to be greatest at a Brinell hardness of 180—210 corresponding to a pearlitic matrix. The effect of manganese on the expansion and constitution of cast iron is shown. Annealing for 1 hr. did not appreciably affect the tensile strength or Brinell hardness up to 650° for 1·44% Si and 600° for 1·69% Si. Quenching in oil and reheating increased the tensile strength. There was no appreciable effect on tensile strength and amount of combined carbon by quenching up to 600°, but above this temperature a rapid fall occurred.

T. H. Burnham.

Fragility of steel. P. Régnauld (Rev. Mét., 1927, **24**, 509—515).—Some varieties of special steels after hardening develop internal fissures on keeping or during use, so that the resistance to shock becomes greatly reduced whereas little change occurs in the tensile strength. This phenomenon is discussed theoretically from the point of view of hidden internal stress and molecular de-cohesion, and it is suggested that the mechanism is analogous to that of season-cracking in brass. A. R. Powell.

Determination of oxide inclusions in pig iron and steel. P. Oberhoffer and E. Ammann (Stahl u. Eisen, 1927, **47**, 1536—1540).—For the determination of silica and silicate inclusions in steel 20 g. of the turnings are dissolved in a slight excess of a cold filtered solution of 100 g. of potassium bromide and 160 g. of bromine in 1 litre of water. The liquid is filtered on a "Cella" membrane filter, the insoluble material washed thoroughly first with cold then with hot water, the gelatinous silica formed by oxidation of any silicide in the steel dissolved by pouring 500 c.c. of hot 3% sodium carbonate solution through the filter, and the thoroughly washed residue treated with 50 c.c. of cold 5% hydrochloric acid and again washed with cold water. The final residue is ignited, weighed, treated with hydrofluoric and sulphuric acids, dried, and again ignited and weighed, loss in weight being equivalent to silica in the steel. The residue contains any alumina originally present; this may be dissolved by fusion with pyrosulphate and recovered as phosphate in the usual way. The bromine method gives low and erratic results for ferrous and manganous oxide inclusions. A. R. Powell.

Paradox of corrosion and protective film [on iron] theory. T. Fujihara (Ind. Eng. Chem., 1927, **19**, 1008—1009; cf. B., 1926, 243).—The "paradox" consists in the fact that the corrosion product being protective, the more rapid the initial corrosion the less the final effect. The comparative rates of corrosion of

Armco iron and electrolytic iron by a drop of distilled water are illustrated by photomicrographs. The corrosion as measured by the diameter of corrosion spots was found with the former to cease after a short time, but with the latter to grow steadily. A similar result with Armco iron was obtained by the oxygen drop method. This less pure iron forms a protective film of corrosion product, whilst the pure electrolytic iron does not.

C. IRWIN.

Silica gel as a medium for drying blast. E. H. LEWIS (Iron and Steel Inst., Sept., 1927. Advance copy. 10 pp.).—The silica-gel drying plant installed at Wishaw to treat 35,000 cub. ft. of air per min. consists of six adsorber units, five being in use while one is being activated by the combustion of blast-furnace gas. The air is supplied at a constant degree of humidity. Comparative data are given for wet- and dry-blast conditions. The output per furnace has been increased by not less than 7½%, and the iron produced has been of more regular quality. There has been no loss of tar with the drier air conditions, and only a slight reduction in ammonia recovery. The costs of running the plant are small.

T. H. BURNHAM.

Behaviour of mild steel under prolonged stress at 300°. W. ROSENHAIN and D. HANSON (Iron and Steel Inst., Sept., 1927. Advance copy. 6 pp.).—Specimens of rolled mild steel containing 0·11% C and 0·39% Mn were heat-treated to give different forms and distribution of cementite and subjected to uniform loads one third, one half, and two thirds of the tensile strength at 300° for over 5 years. In no case did fracture occur, and the actual extension was very slight except in the case of one of the more heavily stressed specimens. The Brinell hardness of the stressed portions of the test pieces increased considerably, much more than would have been anticipated from the amount of work done on the material. No changes in microstructure were observed.

T. H. BURNHAM.

Work-hardening of steel by abrasion. E. G. HERBERT (Iron and Steel Inst., Sept., 1927. Advance copy. 12 pp.).—The work-hardening of metals as measured by the Herbert pendulum increased to a maximum and then fell off. This increase in hardness occurred not only in soft steel, but in hardened steel, whether hardened throughout or case-hardened. The increase in hardness of alloy steels was greater than that of carbon steels. In metals subject to abrasion a surface layer of higher hardness is produced, the maximum corresponding to that induced by the pendulum test, which is thus a measure of the resistance of the metal to wear. When work-hardening occurs, plastic flow of the subjacent metal is caused, even in hardened steel.

T. H. BURNHAM.

Magnetic and other changes concerned in the temper-brittleness of nickel–chromium steels. H. A. DICKIE (Iron and Steel Inst., Sept., 1927. Advance copy. 16 pp.).—Specimens of three nickel–chromium steels were treated to obtain the tough, intermediate, and fully brittle conditions, each bar being tempered for a total of 4 hrs. at 660°. The slow-cooling treatment produced an increase in remanence up to 8% and a rise in maximum permeability. The coercive force and hysteresis loss decreased slightly. The magnetic state of the intermediate condition was similar to that of the brittle condition. The specific resistance decreased with the rate of cooling. The specific volume and hardness were least in the intermediate condition. These features support the view that temper-brittleness is connected with dissolution and redeposition of carbides. On slow-cooling, a portion of the carbide separates out of solid solution in the ferrite, thus modifying the magnetic properties and specific resistance; at moderate cooling rates this also results in a contraction and softening. When cooling is very slow, a network of carbide forms round the grains and brittleness is developed.

T. H. BURNHAM.

Effect of work and annealing on the lead–tin eutectic. F. HARGREAVES (Inst. Metals, Sept., 1927. Advance copy. 12 pp. Cf. B., 1927, 255).—Specimens of the lead–tin eutectic alloy were cast under different conditions of cooling, and were then reduced to varying degrees by hammering. The Brinell hardness was then measured immediately and after keeping for varying periods at room temperature, 100°, and 175°. The hardness is lowered by cold-working, and with severe reduction (78%) in thickness the hardness may become less than that of either constituent of the eutectic. There is a critical range at about 30% reduction, and when this is exceeded the softening becomes much more pronounced. On being kept at room temperature the hardness of the deformed specimens gradually increases, except for those in which the reduction is less than 30%. At 100° there is a general increase in hardness which may be followed by a decrease, whilst at 175° alloys with less than 26·5% reduction return to the hardness of the original casting; those with more than 45% reduction in thickness show a further increase. Results for the silver–copper eutectic are also given.

W. HUME-ROTHERY.

Constitution of alloys of aluminium with silicon and iron. A. G. C. GWYER and H. W. L. PHILLIPS (Inst. Metals, Sept., 1927. Advance copy. 54 pp.).—The equilibrium diagram of the system aluminium–silicon has been investigated by thermal and microscopical methods. The diagram shows a simple eutectic system, the eutectic point being at 11·70% Si and 577—578°. The solubility of silicon in solid aluminium is quite low, remaining almost constant at 0·2% up to 450° and then increasing rather rapidly to about 1% at 550°. Consequently the chill-cast alloys show slight age-hardening. From the eutectic arrest times the solubility of aluminium in solid silicon is probably less than 2%, but this was not determined in detail. The equilibrium diagram of the system aluminium–iron has been similarly determined. Aluminium and the compound $FeAl_3$ form a eutectic at 1·89% Fe and 653°, and there is no solid solution in aluminium. The liquidus rises from the eutectic to a slight maximum at 40·6—41·1% Fe, and alloys in this region are homogeneous ($FeAl_3$). A second maximum occurs on the liquidus at 45·3% corresponding to the compound Fe_2Al_3 with limited solid solubility. This compound forms a eutectic with a constituent denoted *A*, which is of variable composition, but is stable over a limited temperature range only and exhibits a well-defined eutectoid transformation at 57% Fe and 1103°, at which the constituent *A* breaks up into a constituent *B*

containing about 53% Fe, and an iron-rich solid solution *C* which extends from about 65 to 100% Fe. In the unetched state the compound $FeAl_3$ is pale lavender in colour, whist Fe_2Al_5 is Chinese white. The constituent *A* is formed at 1232° by a peritectic reaction between *C* and the liquid. The constitution and structure of the ternary aluminium–silicon–iron alloys over the range 0—20% Fe and 0—30% Si have been studied in detail, a general survey being made of the whole ternary system lying between the aluminium–silicon face of the triangular model and the section parallel to the face comprising the alloys containing 30% Fe. The results are very complex and involve both stable and metastable conditions. In addition to the constituents previously known, namely aluminium, silicon, $FeAl_3$, and *X* (Rosenhain, Archbutt, and Hanson, B., 1921, 851 A), other ternary compounds β, δ, and ζ are present; of these constituents β and *X* are ternary solid solutions.

W. HUME-ROTHERY.

Age-hardening tests with elektron alloys. K. L. MEISSNER (Inst. Metals, Sept., 1927. Advance copy. 18 pp.).—Age-hardening tests have been carried out on various "elektron" alloys containing 0—10% Al and 0—4% Zn, the remainder being magnesium. The specimens were annealed at temperatures of 400—440°, quenched in water, and then tested after keeping for varying periods at temperatures up to 200°. No appreciable age-hardening occurred on keeping the quenched specimens at room temperature. Considerable age-hardening could be obtained by "artificial ageing" at temperatures of 50—200°, provided that the amount of aluminium or zinc, or both metals, present was in excess of the solubility in magnesium at ageing temperature. The solubility of both zinc and aluminium in solid magnesium increases with the temperature, and this accounts for the age-hardening, which is due to the precipitation of particles of the compounds $MgZn_2$ and either Al_2Mg_3 or Al_3Mg_4. A new diagram for the solubility of zinc in solid magnesium is given. W. HUME-ROTHERY.

Grain growth in compressed metal powder. C. J. SMITHELLS, W. R. PITKIN, and J. W. AVERY (Inst. Metals, Sept., 1927. Advance copy. 13 pp.).—Tungsten powders having a mean particle size of from 0·6 to 3·5μ were compressed into bars by pressures of from 8 to 32 tons/in.^{2} When such bars were heated in hydrogen to different temperatures and then allowed to cool, a marked shrinkage took place when the heating temperature exceeded a certain limit. A similar discontinuity was shown by the number of watts required to heat the bar electrically to gradually increasing temperatures. These changes are attributed to grain growth beginning at a certain definite temperature which depends on the size of the initial particles and on the pressure at which the bars were formed; this is confirmed microscopically. Under the above conditions the temperatures at which grain growth began varied from 1100° to 1500° Abs., a low temperature being favoured by small particle size and high pressure. The results are in conflict with those of Sauerwald (A., 1922, ii, 746), who detected no grain growth below 2800° Abs.

W. HUME-ROTHERY.

Influence of rate of cooling on structure of alloys. A. A. BOTSCHWAR (Z. anorg. Chem., 1927, **164**, 189—194).—In order to ascertain the influence of rate of cooling on the pearlitic crystallisation of carbon steels, the structure of steel rods which had been heated at about 100° above the Ac3 point, then chilled at one end, was examined. In steels containing 0·11—0·53% of carbon, the region of transition from the non-chilled to the chilled part, which corresponds with the maximum rate of cooling and the maximum supercooling, consisted of martensite and the eutectoid interspersed with the primary ferrite. Hence, with the maximum supercooling the rate of the primary crystallisation always exceeded that of the eutectic crystallisation. The transition region in steels with 0·6—1·43% C, on the other hand, showed no signs of the primary crystallisation, so that the rate of this process must be equal to or smaller than that of the eutectic crystallisation.

R. CUTHILL.

Cathodic disintegration as a method of etching specimens for metallography. C. S. SMITH (Inst. Metals, Sept., 1927. Advance copy. 3 pp.).—The method of cathodic sputtering can be used to develop the structure of certain alloys for microscopical examination. A simple apparatus is described in which the specimen is attached to the cathode, and a voltage of 2000—7500, depending on the gas pressure, is used. The method is particularly suitable for copper–silver alloys.

W. HUME-ROTHERY.

PATENTS.

Bimetallic element. W. M. CHACE, Assr. to W. M. CHACE VALVE CO. (U.S.P. 1,642,485, 13.9.27. Appl., 6.7.25).—One of the elements consists of a nickel-steel alloy containing not more than 42% Ni to which is welded a steel the composition of which is approximately 64·05% Fe, 22·00% Ni, 8·00% Cr, 2·00% Cu, 1·00% Co, 1·75% Si, 0·70% Mn, and 0·50% C.

F. G. CROSSE.

Aluminium alloy. ÉTAB. MÉTALLURG. DE LA GIRONDE (F.P. 613,758, 11.2.26. Conv., 19.2.25).—The alloy comprises 96·91% Al, 2% Cu, 0·37% Ni, 0·27% W, and 0·45% Mg. The copper, nickel, and tungsten are melted with a small part of the aluminium, and the alloy together with the magnesium are added to the remainder of the aluminium. The product has a high tensile strength and ductility, and remains unaffected by repeated melting. A. R. POWELL.

Compound to solder aluminium. G. E. GAIL, Assr. to A. SEITZ (U.S.P. 1,641,422, 6.9.27. Appl., 12.3.26).—The method of making an aluminium solder consists in melting, chilling, and remelting 9¼ lb. of zinc, and adding to the melt in sequence 15¼ lb. of block tin, ¼ lb. of lead, and ¼ lb. of phosphorous tin. The mixture is stirred and cleared by immersing a block of wood, finally adding ¼ oz. of beeswax to assist in removing impurities. C. A. KING.

Solder for aluminium and its alloys. A. PASSALACQUA (F.P. 611,114, 23.5.25).—A solder for aluminium comprises an alloy of aluminium, tin, zinc, magnesium, manganese, bismuth, cadmium, and cobalt in suitable quantities. The parts to be soldered are heated to 250—550°, and the solder is rubbed on with metal brushes.

A. R. POWELL.

Material for making tools of high mechanical strength. STAHLWERKE RÖCHLING-BUDERUS A.-G. (F.P. 609,734, 24.1.26. Conv., 25.2.25).—Finely-powdered oxides of metals having a high m.p., with or without a proportion of the corresponding metal, are melted in graphite crucibles, in a carbonising atmosphere, or in the presence of metal carbides having a high carbon content, in such a manner that air is excluded from the fused mass. The proportions of the constituents are so arranged that the mixture has the eutectic composition. *E.g.*, 100 pts. of crude tungsten are fused with 5—10 pts. of thorium carbide and 3—5 pts. of molybdenum, and immediately the mixture is molten it is cast into suitable moulds. A. R. POWELL.

Recovery of gold from sea water. B. SZILARD (F.P. 612,081, 18.6.25).—Filtered sea-water is treated with a reagent which precipitates gold from its compounds, then with a second reagent which gives an insoluble compound with the first; *e.g.*, ferrous sulphate and barium chloride may be used. The precipitate is removed and the water evaporated to recover sodium chloride. A. R. POWELL.

Apparatus for production of pure metal. S. L. MADORSKY, Assr. to GATHMYS RESEARCH CORP. (U.S.P. 1,642,683, 20.9.27. Appl., 5.7.24).—A converter is provided with a spout which can be brought into position with either a melting furnace or a discharge duct. The latter is provided with means for preheating a reducing gas before its introduction into the contents of the converter. M. E. NOTTAGE.

Process for recovering metals from slag. F. ROSENZWEIG (U.S.P. 1,643,610, 27.9.27. Appl., 17.9.26).—The blast-furnace slag which contains the metal in the form of an oxide is allowed to flow from the furnace and an electric current applied to it while still molten to reduce the oxide to metal, which is then collected. M. E. NOTTAGE.

Furnace for heating or melting metals. A. ZILLIACUS (U.S.P. 1,643,569, 27.9.27. Appl., 25.3.26. Ger., 28.3.25).—The furnace consists of a hearth on which a layer of protective gas to envelope the metal can be produced, and across which a heating flame can be passed above the protective gas. M. E. NOTTAGE.

Gas-fired annealing furnace. DOWSON & MASON GAS PLANT CO., LTD., and J. PATON (E.P. 277,251, 26.4.27).—In a furnace particularly suitable for annealing malleable castings the furnace chamber is preferably divided into a number of soaking pits, operated independently, by means of hollow transverse walls which contain horizontal passages for heating air to be delivered through ports in the side wall of the furnace. Gas is also conducted through a duct in the furnace wall to ports situated below the air ports. Waste gases leave through ports connecting with a flue in the opposite side wall of the furnace. C. A. KING.

Working bright-annealing furnaces. SIEMENS ELEKTROWARME-GES.M.B.H. (E.P. 272,214, 31.5.27. Ger., 3.6.26).—The protecting gas in a bright-annealing process circulates upwardly through the furnace and downwardly in an adjacent cooling chamber, due to the difference in density between the hot gas in the furnace and the cooling gas. C. A. KING.

Producing a refractory coating on metallic surfaces. PETROLE SYNTHETIQUE SOC. ANON., and A. FOLLIET (E.P. 277,211, 1.1.27).—Metals, *e.g.*, iron and steel, are heated at about 800° in contact with a mixture of aluminium, silica, and a volatile chloride, in a closed container. C. A. KING.

Coating of materials by metal spraying. F. G. COZENS, and METALLISATION, LTD. (E.P. 277,071, 4.6.26).—For coating the interior of elongated articles, *e.g.*, pipes, the metal-spraying jet is carried by a longer extension tube and the issuing metal is deflected at any desired angle by means of an independent blast. The article to be coated is given a rotary and longitudinal motion in relation to the jet. The method may be applied to sand-blasting. C. A. KING.

Protection of metal surfaces. DUNLOP RUBBER CO., LTD., A. LAKEMAN, and F. C. MACCABE (E.P. 276,705, 29.4.26).—A vulcanising solution is applied to the cleaned metal surface, which is then covered with a sheet made by mixing at least 75% of tyre scrap with sulphur, brown substitute, mineral oil, and stearic acid; the whole is then vulcanised, preferably in an elastic mould. M. E. NOTTAGE.

XII.—FATS; OILS; WAXES.

Detergent action of soaps. II. G. P. VINCENT (J. Physical Chem., 1927, 31, 1281—1315).—Soap solutions suspend solid material stably because the solid particles adsorb negative ions from the solution, and cataphoresis experiments show that the manganese dioxide used by Fall (B., 1927, 727) is negatively charged in dilute solutions. Positive ions are also adsorbed and more strongly with increasing concentration until a region of zero stabilisation by the soap is reached. The maximum stabilisation observed by Fall (*loc. cit.*) arises when the negative in comparison with the positive adsorption is greatest. The discrepancy between the results of McBain (A., 1924, ii, 155) and Fall disappears when the carbon suspensions used by the former are so treated that the non-stabilised carbon does not clog the pores of the filter paper used. The emulsification of kerosene oil and a heavy machine oil by palm oil, olive oil, tallow, commercial Green Arrow, and commercial silicated Green Arrow soaps, which vary but little in their emulsifying powers, is at a maximum at a soap concentration of 0·05—0·10%. A concentration higher than this maximum is beneficial, but temperatures above 40°—the optimum—are detrimental. Emulsification is furthered by the presence of ammonia solution (optimum concentration 1·2%), sodium orthophosphate, sodium hydroxide (0·32%), and to a lesser degree by sodium carbonate, and by borax. An excess of sodium hydroxide, but not of ammonia and its salts is detrimental. Contrary to the findings of Stericker (B., 1923, 364 A), sodium silicate, alone or mixed with ammonia, is not an emulsifier of oils (cf. Fall, *loc. cit.*). The importance of wetting in detergency has been studied. Sodium phosphate will cleanse from oils which it does not emulsify. Emulsification is not essential, although highly beneficial. The high detergent value of concentrated soap solutions lies in their efficiency as both wetting and emulsifying agents. Sodium phosphate

is especially efficacious with greases containing calcium oleate on account of the formation of sodium soap from the calcium salt present. The grease is disintegrated and the sodium soap thoroughly incorporated therein. The wetting power of the phosphate is also a factor. A mixture containing 20% of soap and 80% of sodium silicate of the correct composition has noteworthy possibilities as a detergent, and its effect in water softening, content of free alkali, and upon textile fibres has been investigated. Sodium hydroxide is more efficient than sodium silicate in softening waters containing iron, but the carbonate is less so. The beneficial effects obtained by adding sodium silicate to a bleaching solution of sodium hypochlorite are as follow: (i) weakening of the bleaching solution is reduced by forcing back the reaction forming hypochlorous acid; (ii) the cloth fibres are less weakened because of less bleaching, and silica, which strengthens the fibres, is probably adsorbed; and (iii) the removal of the yellow decomposition product formed is facilitated.

L. S. THEOBALD.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

New cadmium pigments. A. EIBNER (Farben-Ztg., 1927, **32**, 2308—2309).—The "cadmopone" pigments, relatively new to England (cf. Ward, B., 1927, 196), have been manufactured in Germany since 1907, and references to the literature are given. The more recent development (1919) in this field is the production of cadmium reds etc. as pure pigments (free from co-precipitated barium sulphate etc.). These consist of cadmium sulphide–selenide mixtures, and their colour, purity of tone, and fastness to light are discussed.

S. S. WOOLF.

Arsenic in printing inks. R. S. MORRELL and C. I. SMYTH (Analyst, 1927, **52**, 339; cf. B., 1927, 371).—The proposed limit for arsenic in printing ink of 1 in 50,000 is considered unnecessary, since it would mean only 1 pt. in 5 million of foodstuff, assuming all the printing ink to be absorbed by the food, or the whole wrapper to be consumed. Further, it would involve the use of arsenic-free chemicals in the manufacture of the ink. One part of arsenic in 7000 of ink would mean 1 pt. in 700,000 of food, and would not exceed the legal limit, even if all the arsenic were absorbed.

D. G. HEWER.

Composition of the white resin of *Pinus silvestris*. B. A. ARBOUSOV (J. Russ. Phys. Chem. Soc., 1927, **59**, 247—264).—Owing to the difficulty of obtaining pure individual products from terpenes, due to their readiness to isomerise, and to their close resemblances among themselves (the hydrogenation of α-pinene is cited), it has been decided to base the classification of turpentine derivatives upon their physical methods of separation and identification, the optical methods of analyses of turpentine showing special significance, especially the measuring of the rotatory dispersion. The several physical methods suggested are critically discussed, the data obtained for the physical constants of the several fractions of the liquid portion of the white resin from *P. silvestris* being tabulated. It is concluded that (1) the white resin obtained without access of air contains 35·5% of turpentine, an amount exceeding that of turpentine in non-treated resin (31%) according to the hypothesis of Dupon; (2) this turpentine contains up to 87% of α-pinene; (3) the prepared α-pinene, with $[\alpha]_D + 40{\cdot}79°$, shows it to be more optically pure α-pinene of *P. silvestris* than that described in the literature; and (4) the chemical characteristics of *d*-α-pinene may depend upon its rotatory dispersion (for this compound, $\alpha_F/\alpha_C = 1{\cdot}97$). Further, the rotatory dispersion may be used not only for the determination of the purity of pinene, but also for that of the turpentine obtained from the resin of *P. silvestris*.

J. KAYE.

Two natural resins. F. H. GEAKE (Ind. Eng. Chem., 1927, **19**, 826—827).—Two new resins from trees growing in south-eastern Siam have been investigated. Neither is completely soluble in alcohol and the solubility is not improved by "melting." Neither resin yields an appreciable quantity of essential oil. Both give varnishes with alcohol, benzene, and oil of turpentine. Both resins yield oils on dry distillation, which, however, do not resemble rosin oil, and are not likely to find immediate commercial application.

A. B. MANNING.

XV.—LEATHER; GLUE.

Chemistry of liming. G. D. MCLAUGHLIN, J. H. HIGHBERGER, and E. K. MOORE (J. Amer. Leather Chem. Assoc., 1927, **22**, 345—363).—It has been shown that 76% of the total sulphur dissolved by a lime liquor is derived from the epidermis, 17% from the corium, and 7% from the hair. A certain minimum degree of degradation of the epidermis must occur before the hair is loosened, and, judged by the amount of dissolved sulphur in the lime liquor, the decomposition of keratin is the same for a given skin or hide, at the point of hair loosening, regardless of the time required to reach this point. Primary alkylamines greatly reduced the time required to produce satisfactory hair loosening in fresh lime liquors to which they had been added, but secondary and tertiary amines had little or no effect. Ammonia added to fresh lime liquor in greater amounts than are found in very mellow lime liquors had no effect on the rate of hair loosening, but only increased the amount of dissolved skin substance. The increased hair-loosening properties of a mellow lime liquor are attributed to the appreciable quantities of amines present. These do not increase the solubility of the lime nor the alkalinity of the liquor, but probably react directly with the epidermal keratin. The inhibitory influence of salt in lime liquors is due to an actual retardation of the decomposition of the epidermal keratin.

D. WOODROFFE.

Bacteriology of liming. G. D. MCLAUGHLIN, G. E. ROCKWELL, and I. H. BLANK (J. Amer. Leather Chem. Assoc., 1927, **22**, 329—344).—Pieces of freshly-flayed steer hide were kept for varying periods of time and limed. The longer the period of keeping, the shorter the time required for hair loosening, the greater the total and non-protein nitrogen in solution, the less the swelling, and the greater the amount of hide sulphur in the liquor. These effects are attributed to the action of bacteria which affect the physical and chemical condition of the

hide before it is limed. Thus the cure of a skin affects the liming and liming effects. Over-soaking affects the liming process in the same way as delay in curing or exposure of fresh hide to air. Liming tests were made with and without the addition of chloroform to water and lime water respectively. Fresh hide unhaired in water undoubtedly as a result of bacterial action because it did not unhair in water and chloroform. The chloroform did not prevent unhairing in lime liquor so that unhairing is possible without the aid of bacterial growth. Non-spore-forming bacteria were killed by fresh lime solution containing excess lime whilst sporulating micro-organisms were not killed. The antiseptic value of lime increases with increasing temperature and increasing alkalinity, the latter being probably due to its ability to remove carbon dioxide, which is essential to bacterial growth. Bacteria do not function in lime liquors under normal conditions but only when the skin entering the liquors contains so many bacteria due to poor curing or over-soaking that the lime liquor is unable to cope with them. A photomicrograph of a section of an over-soaked hide which has been limed reveals numerous bacteria in the depths of the hide. There are two types of mellow limes, one in which only the products of hydrolysis have accumulated, and the other containing in addition the accumulation of the protein cleavage products resulting from bacterial action. D. WOODROFFE.

Micro-tannology of unhairing [hides and skins]. G. D. MCLAUGHLIN and F. O'FLAHERTY (J. Amer. Leather Chem. Assoc., 1927, **22**, 323—329).—Samples of steer, calf, and goat skins were soaked in 4 times their weight of water at 20°, then the steer and calf skin limed in 4 times their weight of lime liquor containing 6% of excess lime on the weight of unsoaked skin. The goatskin was limed in 12 times its weight of lime liquor. Different tests were made with and without additions of ammonia, amines, and sodium sulphide respectively and for different periods. The results show that the main function of an unhairing solution is so to digest the skin epidermis that it, together with the hair or wool, may be easily removed by mechanical means. Unhairing can be effected by any agent capable of digesting, destroying, or detaching the epidermis from the surface and the hair follicle linings of the skin. The gland cells are digested during the process and removed as débris. The usual unhairing agents employed affect the epidermis and glands by digestion and the hair and corium by swelling or plumping, accompanied by more or less dissolution of the skin and hair proteins. If the skin remains in the unhairing solution longer than is necessary for unhairing, an abnormally large amount of the skin is dissolved by the solution, which is harmful for heavy hides. Too short a time is also harmful. The desirable unhairing agent is one that requires the least time and does the least damage to the skin or hair. D. WOODROFFE.

XVI.—AGRICULTURE.

Nitrate assimilation by soil micro-organisms in relation to available energy supply. F. E. ALLISON (Soil Sci., 1927, **24**, 79—93).—Biological activity in soil is stimulated by the addition of easily-decomposable organic matter, and the increased utilisation of nitrates by the organisms may result in injury to growing crops, due to lack of nitrate supplies. The injury may be prevented by treatment with sufficient sodium nitrate. The extent of the nitrate starvation depends upon the carbon : nitrogen ratio of the material added. The unfavourable effect observed when fresh farmyard manure is used is attributed solely to this cause. Soil so treated recovers as the stored-up nitrogen in dead bacterial cells becomes again available to the plant. A. G. POLLARD.

Delayed effect of liming [soils]. P. E. KARRAKER (Soil Sci., 1927, **24**, 147—148).—A period of some weeks was observed between the application of limestone to an acid soil and the resulting improvement in the growth of lucerne. The importance of this delayed action in experimental work is emphasised. A. G. POLLARD.

Solodisation (dealkalisation) of soils. K. K. GEDROIZ (Nosovskaya Selskokhosyaistvennaya Opuitnaya Stantzia [Russia], Bull., 1926, No. 44, 1—48).—There are essential differences between the degraded alkali soils (solonetz) and the primary podsols. A degraded solonetz is the final stage in the evolution of the salinised soils ; the destructive effects on the humus and the aluminosilicate complex which are highly dispersed result in the formation of compounds, *e.g.*, amorphous silica, not present in podsols. In alkali soils not containing calcium carbonate, dealkalisation ("solodisation") is more rapid ; the inactive portion of the humus remains, and the silica content is increased. CHEMICAL ABSTRACTS.

Effect of acidity on the activities of the micro-organisms of the soil. E. H. PANGANIBAN (Sugar News, 1926, **7**, 902—905).—Ammonification is only slightly affected whilst nitrification is greatly depressed by soil acidity. p_H 6 is about the most acid limit for the activity of nitrifying organisms. Denitrification and sulphofication are also affected by soil acidity. CHEMICAL ABSTRACTS.

Nitrogen manuring necessary for sugar-beet grown after lucerne, red clover, or after green-manuring and farmyard manure. SCHÖNBRUNN (Z. Pflanz. Düng., 1927, **6B**, 399—404).—Following lucerne or red clover, sugar-beet needs no nitrogenous fertiliser except possibly a light top-dressing to give the young plants a start. After straw crops moderate dressings of farmyard manure should be supplemented by some nitrogen fertiliser. Following a green manure crop and a moderate dressing of farmyard manure artificial nitrogenous fertilisers should only be necessary for sugar-beet when the green manure crop was one of low-nitrogen content. A. G. POLLARD.

Influence of potash manures in conjunction with increasing supplies of nitrogen on the yield and quality of barley in 1926. H. WIESSMANN and K. BÜRGER (Z. Pflanz. Düng., 1927, **6B**, 408—417).—Potash manures were profitable where moderate supplies of nitrogen were available in the soil. Heavy manuring with ammonium sulphate was not profitable either alone or when used in conjunction with potash fertilisers.

Neither nitrogenous nor potash fertilisers appeared to influence the protein content of barley.

A. G. Pollard.

Stimulation [of plant growth]. J. H. Aberson (Z. Pflanz. Düng., 1927, **6B**, 405—407).—Mixtures of magnesium and manganese salts were used as stimulating agents on a number of common farm crops. No positive results were obtained. A. G. Pollard.

Membrane filters in soil analysis. W. Hoffmann (Z. angew. Chem., 1927, **40**, 1052—1058).—The use of membrane filters for the determination of silica in soils is cheaper and quicker than the ordinary process involving a double evaporation with hydrochloric acid and heating at 130°. In practice, 50—100 g. of the ignited soil are digested with 125 c.c. of 20% hydrochloric acid and 3—5 drops of nitric acid for 1—2 hrs. After cooling for 1½ hrs., 300 c.c. of water are added and the whole is filtered under reduced pressure through a membrane filter and washed with 5% hydrochloric acid. The silica and insoluble material are removed from the filter, dried, and ignited, and the other constituents are determined in the filtrate in the usual manner. An 80—100 sec. membrane filter of pore size 0·2—0·3 μ is most suitable. The complete filtration occupies ¾—1 hr., and the amount of silica passing into the filtrate is only 0·03—0·05%, as compared with about 0·02% in the double-evaporation method. By using finer filters, or by a preliminary evaporation to dryness, the silica remaining in solution may be still further reduced, but the time required is increased in both cases. Addition of more than a few drops of nitric acid increases the proportion of silica passing into the filtrate.

F. R. Ennos.

Changes in reaction with the ageing of soil samples. B. Aarnio and A. Salminen (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1927, **B**, 30—33).—Determinations of the p_H value of soils must be made with fresh field samples. Drying of soils in the laboratory largely increases their active acidity. This is particularly the case with soils rich in humus.

A. G. Pollard.

Effect of drying on different types of soils in the "tchernozem" and "podsol" zones of European Russia. A. Lebediantzev (Compt. rend., 1927, **185**, 568—569).—The increased fertility of soils on drying is a common property of the types examined.

H. Burton.

XIX.—FOODS.

Constants of cow ghee. B. B. Brahmachari (Indian Med. Gaz., 1927, **62**, 318—322).—The Reichert–Wollny value is 19·52—42·35 c.c. (weighted average 25·3 c.c.); butyro-refractometer reading 39·6—44·6 at 20°; saponif. value 213·9—236·4 (average 224·4); iodine value 25·6—41·1 (average 35·8).

Chemical Abstracts.

Determination of sulphur dioxide in dried fruit. P. May (Analyst, 1927, **52**, 526; cf. Miller, B., 1927, 615). —Although Miller's method accurately determines the total sulphur dioxide in the distillate, it is not a measure of that in the fruit since prolonged boiling with fairly strong acid is necessary to liberate all the original sulphur dioxide. Miller's method should be useful for quick sorting purposes. D. G. Hewer.

Metabolism of nitrogen compounds in dormant and non-dormant potato tubers. W. Newton (J. Agric. Res., 1927, **35**, 141—146).—Sand culture experiments show that absorption of nitrates by the tissues of potato tubers decreases the dormant period. This effect is not produced by ammonium salts. Amino- and amide-nitrogen in non-dormant tubers is usually greater than in dormant ones, but growth is not dependent on the actual concentration of either of these. Examination of the expressed juice of dormant and non-dormant tubers shows the proteolytic enzyme activity to be greater in the latter. Following the addition of casein to the expressed juice, the rate of accumulation of amino-acid becomes a direct function of the period of incubation. Incubation of the expressed juice results in the conversion of amino-acid nitrogen into amide-nitrogen. Carbohydrate hydrolytic enzymes in dormant potatoes are stimulated by asparagine. A. G. Pollard.

Copper as an industrial contaminant in foodstuffs. C. G. King and G. Etzel (Ind. Eng. Chem., 1927, **19**, 1004—1005).—Down to 5 to 10 mg. of copper per day may produce toxic results. In addition, the metal accelerates the destruction of vitamin-*C*. Four groups of fruit preserves tested were found to contain from 3 to 110 parts per million of copper. The pasteurisation of milk in copper apparatus only added 0·1 p.p.m. to the copper content. Condensed milk condensed in copper vacuum pans averaged 3·7 p.p.m.; carbonated fruit beverages were found normally to contain very little copper. The variations in the fruit preserve samples were apparently due to variation in care between different manufacturers. Copper was determined colorimetrically as xanthate. C. Irwin.

Behaviour of the anthocyan pigments in canning. C. W. Culpepper and J. S. Caldwell (J. Agric. Res., 1927, **35**, 107—132).—Discoloration of fruit and vegetables in tinned containers is limited to those commodities having anthocyan pigments in the skin or flesh. Discoloration is most prominent in fruit well coloured during ripening by sunlight and high temperature, and is traced to interaction between the pigment and the tin of the container. Red anthocyan colours form purple tin salts, the transformation being favoured by alkaline conditions. The action is reversible, hence discoloration is minimised in highly acid fruits. When the inner surface of the container is enamelled discoloration is reduced as the area of attack is smaller. On the other hand, perforation of the tin is hastened as the action is localised. A. G. Pollard.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Patents.

Manufacture of alkyl carbonates. H. G. Mitchell, Assr. to U.S. Industrial Alcohol Co. (U.S.P. 1,638,014, 9.8.27. Appl., 14.7.24).—Producer gas is passed with chlorine over activated charcoal, and the gaseous mixture of carbonyl chloride and nitrogen obtained is treated with alcohol to form ethyl chloroformate and, finally, ethyl carbonate. T. S. Wheeler.

Production of organic [nitrogen] compounds. K. F. SCHMIDT and P. ZUTAVERN, Assrs. to KNOLL & Co. (U.S.P. 1,637,661, 2.8.27. Appl., 11.12.25. Conv., 11.4.25).—Hydrocarbons or their carbonyl derivatives are treated with hydrazoic acid in presence of catalysts, *e.g.*, metallic chlorides, to give nitrogen compounds, *e.g.*, amines or nitriles. Thus a benzene solution of hydrazoic acid is treated with aluminium chloride to form aniline; benzaldehyde gives benzonitrile.
T. S. WHEELER.

Manufacture of camphene. I. G. FARBENIND. A.-G., Assees. of V. HILCKEN (G.P. 439,695, 11.11.24).—Bornyl chloride, or oil containing it, is heated at 190—200° with an aqueous solution of a zinc salt (zinc acetate) in presence of zinc oxide or hydroxide and sodium acetate, which prevent the condensing action of the zinc chloride formed in the reaction. Camphene, in 98% yield, is distilled off in steam, and the aqueous residue, after addition of sodium hydroxide and removal of solid sodium chloride, may be used for a fresh batch. Zinc chloride may be used in place of zinc acetate, and the sodium acetate may be omitted. C. HOLLINS.

Manufacture of coumarin from o-coumaric acid. —I. G. FARBENIND. A.-G., Assees. of K. SCHAFFGANZ (G.P. 440,341, 6.7.24).—The yield of coumarin from coumaric acid (hydrobromic or sulphuric acid gives 30%) is increased to 75% by using a small amount of a mercury salt (mercuric sulphate) alone or with mineral acids, much less tar being formed. Aqueous or dilute alcoholic media may be used. C. HOLLINS.

Manufacture of *o*-aminocinnamic acid. I. G. FARBENIND. A.-G., Assees. of K. OTT and K. SCHAFFGANZ (G.P. 440,052, 13.5.24).—*o*-Cyanocinnamic acid, m.p. 137°, is hydrolysed with hydrochloric acid at 90—95° to *cis-o*-carbamidocinnamic acid, m.p. 239, which is converted by alkaline hypochlorite at 80° into *trans-o*-aminocinnamic acid, m.p. 158°. C. HOLLINS.

Stabilisation of chlorinated hydrocarbons used in cleaning and scouring. J. M. G. DE SCHACKEN (F.P. 615,800, 8.5.26).—The decomposition of tetrachloroethane, trichloroethylene, or the like on exposure to the sunlight may be prevented and the cleansing power increased by addition of 10—20% of toluene, xylene, tetrahydronaphthalene, hydroterpene, or α-dichlorohydrin. A. R. POWELL.

Method for decreasing the toxic action of cocaines. R. ECKERMANN (E.P. 267,463, 25.6.26. Conv., 13.3.26).—By treating cocaine (or its derivatives) with esters of carbamic acid (or their derivatives), both components being first treated with an organic acid, base, mono- or poly-hydric alcohol, or phenol volatile alone or in steam below 125°, products are obtained having decreased toxic action and increased anæsthetic action, and which may be heated at 125° without decomposition. B. FULLMAN.

Manufacture of water-soluble protein-silicic acid compounds. J. A. VON WÜLFING (G.P. 439,043, 3.12.24).—Compounds valuable in place of sodium silicate against arteriosclerosis, carcinoma, and tuberculosis, are obtained by adding a solution of sodium metasilicate (or polysilicate with enough sodium hydroxide or sodium-protein to form metasilicate) to an alcoholic suspension of acid proteins, *e.g.*, nucleoalbumins or albuminates. Compounds of sodium metasilicate with casein and lactalbumin are described.
C. HOLLINS.

XXIII.—SANITATION; WATER PURIFICATION.

Analysis of insecticides containing finely-divided arsenic, borax, or *p*-dichlorobenzene. M. FRANÇOIS and L. SÉGUIN (Ann. Falsif., 1927, 20, 217—224).—The constitution of a number of commercial rat-, mole-, and bettle-killers has been investigated. Those containing free arsenic are usually in the form of deep-coloured powders or pellets, and contain, in addition, a bait such as sugar and an absorbent such as sawdust, kieselguhr, or powdered pumice. Arsenic can generally be detected under the microscope and identified by sublimation in a narrow tube after separation of the soluble matter by washing. The metalloid can be determined by oxidation with concentrated nitric acid (vegetable constituents are destroyed at the same time), diluting, rendering just alkaline with ammonia, and filtering. To the filtrate are added magnesium chloride solution and ammonia, and after 3 days the precipitate of magnesium ammonium arsenate is collected, washed, dried, and weighed. To detect the presence of soluble arsenic compounds, the use of which is illegal, the material is allowed to remain for 30 min. with two portions of cold water, the mixture filtered, and the filtrate evaporated to dryness. The residue is heated with strong nitric acid for 3 hrs. on a brine bath to destroy the sugar etc., and the residue, if any, analysed for arsenic as before. Arsenious and arsenic oxides are differentiated by the colour of the precipitate which the cold water extract gives with silver nitrate solution. Beetle-killers were found to consist usually of borax with a blue colouring matter, the former being readily detected by borax bead tests with cobalt nitrate or calcium fluoride. The colouring matter may be either ultramarine, indigo, or Prussian blue. These are distinguished by treatment with oxalic acid solution in the presence of lead acetate paper. Ultramarine dissolves giving a turbid solution and stains the lead acetate; indigo gives no reaction; whilst Prussian blue dissolves to form a blue solution, but does not colour the acetate paper. Moth preventatives were found to consist of *p*-dichlorobenzene either in solution or as pellets. This substance can be identified by its odour, by the production of hydrochloric acid when mixed with camphor and ignited, by its m.p. and b.p., or by its crystalline form after sublimation in a wide tube.
H. J. DOWDEN.

PATENTS.

Antiseptic and disinfectant. P. L. V. JAMOTTE (E.P. 259,233, 1.10.26. Conv., 1.10.25).—A stable bactericidal *compound* of dioxymethylene and bromine is prepared by oxidation in the cold of methyl alcohol with a solution of bromine in 100 vol. hydrogen peroxide, in the presence of vanadium oxychloride and mercury as catalysts. The addition of a small quantity of formaldehyde hastens the reaction. B. FULLMAN.

Alumina coagulant (U.S.P. 1,643,962).—See VII.